

DEVELOPMENT AND EVALUATION OF COATINGS FOR  
THE PREVENTION OF SURFACE OXIDATION AND  
DECARBURIZATION OF FERROUS MATERIALS  
DURING HEAT TREATMENT

A Thesis Submitted  
in Partial Fulfilment of the Requirements  
for the Degree of  
**MASTER OF TECHNOLOGY**

By  
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to the

**DEPARTMENT OF METALLURGICAL ENGINEERING**  
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**JANUARY, 1979**

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### CERTIFICATE

Certified that the thesis entitled 'Development and Evaluation of Coatings for the Prevention of Surface Oxidation and Decarburization of Ferrous Materials During Heat Treatment' has been carried out under our supervision and the same has not been submitted elsewhere for a degree.

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### ACKNOWLEDGEMENT

I express my deep sence of gratitude to Dr. P.C. Kapur and Dr. Raj Narayan for their valuable suggestions and spirited efforts in bringing this work a success.

I thank B. Sharma, R.K. Prasad, O.P. Malviya, S.R. Chaurasia and A.C. Basak for helping me at various stages of my experimental work. I thank also M.N. Mungole for helping me.

I will take this opportunity to thank all of my friends who helped me. Special mention has to be made of P.K. Ghosh, Pallabsarkar, R.N. Chatterjee, Ravi, Devraj and B.P. Rao. I will also thank D.U. Krishna Rao for his valuable discussions with me, J. Subramanyam and B. Nageswar Rao for helping me in proof reading.

I thank V.P. Gupta for his neat tracings and M.R. Nathwani for his neat typing.

K. Hari Prasad.

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## ABSTRACT

To prevent surface oxidation and decarburiization during heat treatment of ferrous materials the controlled atmosphere furnaces and salt baths are conventionally used. They, however, suffer from many inherent disadvantages e.g. high capital cost of the equipment, high cost of salts and inert gases, explosion hazard etc. Most of these disadvantages can be overcome by using surface coatings. Ideally such a coating should meet the following requirements; It should 1) prevent oxidation and decarburiization, 2) be easy to apply, 3) be cheap, 4) not hinder heat treatment process, 5) have good thermal conductivity and 6) come off easily after heat treatment process.

Two coating compositions, meeting the above requirements, have been developed after extensive trials. These coatings are able to reduce the oxidation loss of ferrous materials by 65% to 98%.

## CHAPTER I

### LITERATURE REVIEW

#### 1.1 INTRODUCTION

Heat treatment is an operation or a combination of operations involving the heating and cooling of metals in the solid state for:

- i) hardening and strengthening of metals,
- ii) relieving internal stresses, as in castings,
- iii) softening objects for further working,
- iv) impregnating the metal surface with C, N etc.,
- v) obtaining combination of strength and useful ductility,
- vi) obtaining desired grain size,
- vii) obtaining desired mechanical properties.

Table 1.1 list various heat treatments normally given to ferrous materials.

#### 1.2 PROBLEMS DURING HEAT TREATMENT:

The properties of any material depend on the characteristics of the bulk and those of the surface. Any change in surface characteristics affects a wide variety of material properties. When steels are heat treated in a furnace in the presence of air and combustion products, surface oxidation and

Table 1.1

HEAT TREATMENTS NORMALLY GIVEN TO FERROUS METALS  
 (Ref. 1 & 2)

S.No.	Material	Heat Treatment	Temp. Range	Atmospheres or salt Baths commonly used.
1.	Carbon and low alloy steels	Annealing	740-930	(i) Nitrogen (ii) Dissociated ammonia (iii) Exothermic gas mixture e.g. 15% $H_2$ ; 10% $CO$ ; 5% $CO_2$ ; 1% $CH_4$ ; and 69% $N_2$
		Tempering		(i) Endothermic generator gas e.g. 4-5% $CO_2$ ; 10-12% $CO$ ; 15-18% $H_2$ ; a trace to 1% Methane and oxygen
		Martempering	40°C above austenitization temperature holding at just above $M_s$ or just below $M_s$ temp.	Molten salt baths.e.g. mixture of nitrates such as potassium nitrate and sodium nitrate
		Austempering	Slightly above $M_s$ temp.	Molten Salt Baths $NaCl$ 45-55% + $KCl$ 45-55%

Table 1.1 Contd.

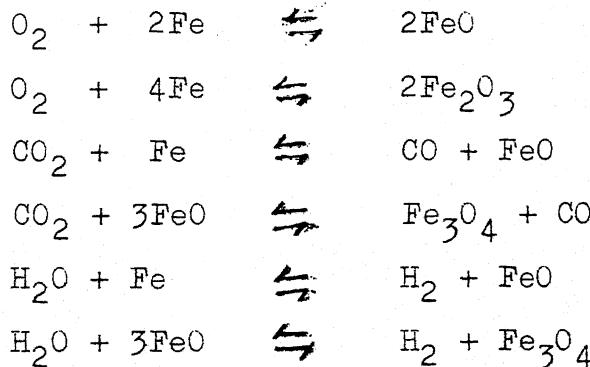
Sl. No.	Material	Heat Treatment	Temp. Range °C	Atmospheres or salt baths commonly used
2.	Tool Steel	Normalizing	763 to 872	(i) Molten lead (i) Ammonia base atmospheres (ii) Endothermic base atmospheres
		Annealing	-do-	-do-
		Austenitizing	763 - 942	(i) Molten Salt baths e.g. Barium chloride, $\text{NaCl}$ , $\text{CaCl}_2$ , $\text{KCl}$ , etc. nitrates.
		Tempering	121 - 482	-do-
3.	Stainless steel	Annealing	1010 to 1121	(a) Cracked ammonia (b) Dry purified hydrogen
	Austenitic stainless steel			
	Ferritic stainless steel	Annealing	649 to 816	-do-
	Martensitic Stainless Steel	Austenitizing	927 to 1066	-do-
		Annealing	649 to 760	-do-
		Tempering	149 to 371	-do-

Table 1.1 Contd.

Sl No.	Material	Heat Treatment	Temp. Range °C	Atmospheres or salt baths commonly used
4.	High Speed tool steel	Annealing	815 - 900	(a) Cracked ammonia (b) Dry purified hydrogen
		Austenitizing	1204 - 1302	-do-
		Tempering		
5.	Carburized Steel	Single Quench	760 to 872	-do-
		Double Quench	After single quench, again heat it to 871	-do-
		Tempering	148 to 205	-do-

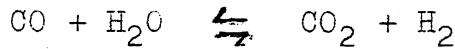
decarburization occurs. Due to oxidation, surface of the components becomes rough and dirty. Apart from metal loss and change in dimensions, this would necessitate cleaning and machining after the heat treatment is over. However, machining may be difficult after certain heat treatment, such as hardening. The carbon content at the surface of the components also decreases due to decarburization. It would then be difficult to achieve the desired mechanical properties at the surface since these properties largely depend on the carbon content of the steel. Therefore all attempts are made to minimize oxidation and decarburization during heat treatment.

OXIDATION: Oxidation of steel is caused by oxygen, carbon dioxide and/or water vapour as in the following general reactions (1,2)

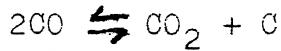


The degree of oxidation or scaling that will be produced, determined by the equilibrium relationship between

iron and iron oxide in contact with CO and  $\text{CO}_2$  or  $\text{H}_2$  and water vapour at the heat treating temperatures. The inter-relation of these four gases is known as water gas reaction<sup>(3)</sup> which may be expressed as:



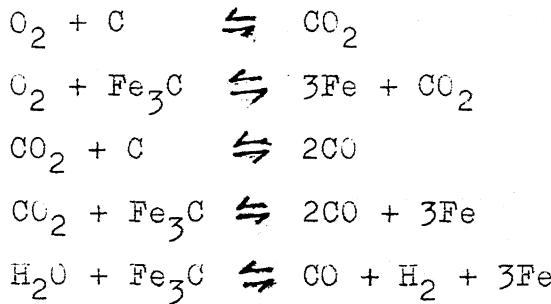
Since furnace atmospheres tend to adjust themselves at operating temperatures, the above reaction is important. If the furnace atmosphere is too high in CO and low in  $\text{H}_2\text{O}$  another reaction known as producer gas reaction<sup>(3)</sup> may take place.



At elevated temperatures steel acts as a catalyst for this reaction which results in the etching or oxidation of bright surfaces and the deposition of soot.

Oxidation of steel may vary from a light adherent straw coloured film which forms at a temperature of about  $180^{\circ}\text{C}$  to a loose blue - black oxide scale that forms at temperatures above  $425^{\circ}\text{C}$ .

DECARBURIZATION: Decarburization of steel surface occurs upon heating steel to a temperature above  $650^{\circ}\text{C}$  and proceeds to greater depths below the surface as a function of time, temperature and furnace atmosphere in accordance with the following typical reactions<sup>(3,4)</sup>:



The equilibrium relationship of these reactions are influenced by the ratio of CO and CO<sub>2</sub> which will be neutral to a given carbon content at a given temperature.

### 1.3 PRESENT METHODS FOR PREVENTING OXIDATION AND DECARBURIZATION DURING HEAT TREATMENT:

To overcome the problems of oxidation and surface decarburization of the ferrous components following methods have been used:

- i) Removal of the oxide scale and decarburized surface by machining after heat treatment.
- ii) Applications of an electroplated coating (12.7  $\mu$  - 25.4  $\mu$ ). Usually of copper, prior to heat treatment.
- iii) Heating parts in a sealed steel box packed in charcoal or cast iron chips,
- iv) Use of molten salt baths as heating media,
- v) Use of protective furnace atmospheres.

By removing the decarburized surface by machining the metal loss during heat treatment can not be prevented, and may not be a desirable remedy in most cases.

By electroplating copper on steel the oxidation and decarburization can be prevented. This is, however, not economical since copper is a costly metal and its removal after the heat treatment might create problems. Moreover, copper itself gets oxidized in some cases<sup>(3)</sup>.

By sealing the parts to be heat treated in steel box, the parts can be heat treated without oxidation and decarburization. But it is not a convenient solution to the problem. Apart from the extra operations of packing, removing and cleaning the parts, the steel container will oxidize and disintegrate. This would necessitate its replacement after few heat treatment cycles.

Rapid heat transfer for austenitizing and tempering semifinished or finished machine parts will be provided by molten salt baths with adequate protection against oxidation and decarburization, provided the composition of the bath is regularly controlled. Certain limitation<sup>(2,3,4)</sup> such as

- i) availability of salts,
- ii) salts are mildly oxidizing and decarburizing to steels at heat treatment temperatures,
- iii) periodic rectification of salts is necessary,

- iv) limitations on operating temperature range,
- v) the difficulty of quenching parts with blind holes etc. restrict the use of salt baths for heat treatment.

At present the application of protective furnace atmospheres is widespread for heat treatment of steels.

Typical protective atmospheres used in industry for preventing oxidation and decarburization are<sup>(1-3)</sup>

- i) Hydrogen,
- ii) Nitrogen,
- iii) Argon,
- iv) Helium,
- v) Dissociated ammonia,
- vi) Exothermic gas,
- vii) Endothermic gas

Table 1.2 shows the gases generally found in protective atmospheres and their reaction with iron and iron oxide.

Some of the draw backs of the protective atmosphere furnaces are<sup>(2-10)</sup>.

- i) Initial cost of the equipment needed is high,
- ii) Cost of the gases used is usually very high,
- iii) They have strong decarburizing potential,
- iv) They are explosive in contact with air,

Table: 1.2

GASES COMMONLY FOUND IN PROTECTIVE FURNACE ATMOSPHERES AND  
THEIR REACTIONS WITH IRON AND IRON CARBIDE <sup>(3)</sup>

Gas	Reactions		
	The gas combining with	Iron	Reaction
Nitrogen		Neutral	
Oxygen	Iron	Iron Oxide	Strongly Oxidizing
	Iron Carbide	Iron	Strongly Decarburizing
Carbon Dioxide	Iron	Iron Oxide	Strongly Oxidizing
	Iron Carbide	Iron	Strongly decarburizing
Water Vapour	Iron	Iron Oxide	Oxidizing
	Iron Carbide	Iron	Strongly Decarburizing
Hydrogen	Iron oxide	Iron	Strongly reducing
Carbon Monoxide	Iron	Iron Carbide	Strongly Carburizing
	Iron Oxide	Iron	Reducing.
Methane	Iron	Iron Carbide	Strongly Carburizing
	Iron Oxide	Iron	Reducing

- v) They could result in hydrogen embrittlement of certain steels,
- vi) They tend to damage thermocouples and decrease the life of the furnace resistors.
- vii) Atmospheres containing CO may cause rapid and serious disintegration of refractory brick work, etc.

#### 1.4 SURFACE COATINGS FOR PREVENTING OXIDATION AND DECARBURIZATION DURING HEAT TREATMENT

Apart from the many disadvantages listed earlier, associated with salt bath and protective atmosphere furnaces, it may be either difficult, when the component size is too large, or uneconomical, when the frequency of use is less, to use these furnaces. In such cases use of a protective coating which can be directly applied on the component surface would be ideal.

The advantages of using such a coating will include the following:

1. It will be possible to use ordinary furnaces without any modification.
2. There will be no need to observe additional safety precautions which have to be normally observed in maintaining the protective atmosphere and salt bath furnaces.
3. There will be no explosion hazard.

4. The life of the thermocouples and furnace resistors will be increased.

Such a coating ideally should:

1. be able to prevent surface oxidation and decarburization of the component.
2. be cheap.
3. be easy to apply.
4. easily come off from the component surface after the heat treatment is over.
5. have good thermal conductivity.
6. not hinder with the normal heat treatment cycle for the given metal.

In a British patent<sup>(11)</sup> it has been claimed that the oxidation rate of steel billets at  $1280^{\circ}\text{C}$  was reduced from  $180 \text{ mg/cm}^2$  to  $20 \text{ mg/cm}^2$  when they were coated prior to heat treatment with a mixture of polyvenylacetate with water containing powders of aluminium and a divalent metal oxide such as  $\text{ZnO}$ ,  $\text{MgO}$ . Using sodium-alluminate  $\text{Cr}_2\text{O}_3$ ,  $\text{SiO}_2$ , sodium polycrylate and water as coating composition Higuchi et. al.<sup>(12)</sup> succeeded in decreasing the oxidation rate of 9% Ni steel from 371  $\text{mg/cm}^2$  to  $171 \text{ mg/cm}^2$  at  $1200^{\circ}\text{C}$  for 3 hrs. Several other investigators<sup>(13-25)</sup> have also claimed that coatings containing  $\text{Al}_2\text{O}_3$ ,  $\text{SiO}_2$ ,  $\text{Na}_2\text{O}$ ,  $\text{B}_2\text{O}_3$ ,  $\text{CaO}$ ,  $\text{MnO}$  etc with different carriers of powders such as silicates, phosphates, bakelite, dextrin

etc. reduce the oxidation and decarburization of steel during heat treatment. No information was, however, available regarding the actual composition, oxidation rate and other desirable coating properties listed earlier.

## CHAPTER II

### EXPERIMENTAL

#### 2.1 MEASUREMENT OF OXIDATION LOSS

Oxidation loss at different temperatures was determined using specimens of mild steel, stainless steel and tool steel (Table 2.1). Surface oxide from the specimens was removed by using a suitable cleaner (Table 2.1).

The raw materials used for coating were ground in a ball mill, for 12 hours using 1/2" and 1" dia balls. Sieve analysis before and after grinding is given in Table 2.2.

20 gm of the coating powder was mixed with 12 cc of the binder into a smooth paste. This was then applied to the cleaned specimen surface using a paint brush.

The coated specimens were air dried and charged in the furnace either at room temperature or at elevated temperature to determine the oxidation loss.

Any loose oxide on the specimen surface, after exposure at high temperature, was first cleaned. If necessary the specimens were cleaned by pickling.

#### 2.2 DECARBURIZATION

Coated and uncoated specimens of 0.56% C steel (Table 2.1) were charged in the furnace at  $1000^{\circ}\text{C}$  for 10 hours followed by air cooling. Specimen cross-section was prepared for metallographic examination.

Table 2.1

**MATERIAL'S CHEMICAL COMPOSITION AND THE PICKLING AGENTS USED**

Sl. No.	Material	Chemical composition weight percentage	Pickling solution composition	Metal attack in 1 minute mg/cm <sup>2</sup>
1	Mild steel	C-0.2, Si-0.11, P-0.02, S-0.01, Mn-0.72	1:1 HCl acid	0.04
2	Stainless steel	C-0.1, Cr-1.9, Ni-11.4	5 by vol. HF+ 20 by vol. HNO <sub>3</sub> + 75 by vol. water	Nil
3	Tool steel	C-0.7, Cr-4.2, V-1.0, Mo-0.33, W-6.0	1:1 HCl acid	Nil
4	Carbon steel	C-0.56, Si-0.11, P-0.02, S-0.01, Mn-0.72	1:1 HCl acid	0.04

Table 2.2

## SHIEVE ANALYSIS OF MATERIALS BEFORE AND AFTER GRINDING

Mesh	A wt%		B wt%		C wt%		D wt%		E wt%	
	No grinding	No grinding	Before grinding	After grinding	Before grinding	After grinding	Before grinding	After grinding	Before grinding	After grinding
- 10 + 60	-	-	-	160.0	-	-	50.0	-	2.0	-
- 60 +100	-	-	-	-	-	-	10.0	-	2.0	-
-100 +140	-	-	-	-	-	-	10.0	-	28.0	-
-140 +200	-	-	-	-	-	-	10.0	-	42.0	-
-200	-	-	-	-	-	-	20.0	-	26.0	-
-200 +270	-	-	-	-	-	-	9.0	-	7.0	-
-270 +325	-	-	-	-	-	-	6.0	-	5.0	-
-325 +400	-	-	-	-	-	-	6.0	-	13.0	-
-400	100.0	100.0	-	-	78.0	-	-	75.0	-	90.0
	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

Surface turnings in steps of 0.127 mm upto a depth of 1.143 mm were removed for chemical analysis.

### 2.3 THERMAL CONDUCTIVITY OF THE COATING

Thermal conductivity of the coating was estimated by measuring the rise in core temperature of 25 cm diameter mild steel bar. The coated and uncoated specimens were charged at room temperature and the furnace temperature was allowed to rise to 1000°C. Good thermal conductivity of the coating would be reflected by a time lag in core temperature rise of coated specimen.

### 2.4 EFFECT OF COATING ON THE HARDENING TREATMENT

Coated and uncoated 0.56 %C steel specimen (Table 2.1) were austenitized at 1000°C for 1 hour followed by water quenching. Specimen cross-section was prepared for micro-hardness measurement.

## CHAPTER III

### RESULTS AND DISCUSSION

#### 3.1 EFFECT OF BINDER COMPOSITION

It was evident from the literature survey that the nature of the binder has an important influence on the coating properties. Several materials were therefore evaluated for their suitability as binders (Table 3.1). Minimum oxidation loss of mild steel at  $800^{\circ}\text{C}$  for 5 hrs was observed when binder 'S' having a specific gravity of 1.2 gm/c.c. was used (Figure 1, Table 3.2).

#### 3.2 EFFECT OF COATING COMPOSITION AND SPECIFIC GRAVITY OF THE BINDER

Initially several coating compositions were tried using binder 'S' having a specific gravity of 1.3 gm/c.c. The results showed that whereas the oxidation loss of uncoating mild steel specimen at  $1000^{\circ}\text{C}$  for 5 hrs was 152 mg/cm<sup>2</sup>, it was possible to reduce it upto a value of 24.035 mg/cm<sup>2</sup> using coating composition GC3 (Table 3.3, Table 3.4). However during the coarse of the investigation it became evident that the specific gravity of the binder 'S' was having an influence on the coating properties. In order to determine the optimum value, the specific gravity of the binder was changed between 1.1 and 1.4 gm/c.c. Using coating composition

Table 3.1

BINDER CODE AND THEIR PREPARATION

Binder Code No.	Method of Preparation
S	Either 60 gms or 75 gms of the binder 'S' were dissolved in 100 cc of boiled water to get binder solution having a specific gravity of 1.2 gm/cc or 1.3 gm/cc respectively.
A	30 gms of binder 'A' of -270 mesh was ball milled with 140 gms of 85 orthophosphoric acid for 1 hr. Then 30 gms of binder 'A' was added and ball milled for 2 hrs. 150 gms of water was added and milled for 15 mins. after 2 hrs. milling. Then the slurry was kept 42 hrs to settle in a beaker. 2.4 gms of $\text{Cr}_2\text{O}_3$ in 20 ml of water was added to the settled mass after clearing the clean solution over the settled mass.
P	4 gms of binder 'P' was dissolved in 100 cc of warm water.

Table 3.2

EFFECT OF BINDER COMPOSITION ON THE OXIDATION  
LOSS OF MILD STEEL AT 800°C FOR 5 Hrs USING  
COATING GC 4\*

Sl. No.:	Binder	Code	Oxidation Loss ( mg/cm <sup>2</sup> )
1.		S	3.94
2.		A	24.11
3.		P	38.28

\* In all cases the coated specimens were air dried for 6 hrs and charged in the furnace at room temperature.

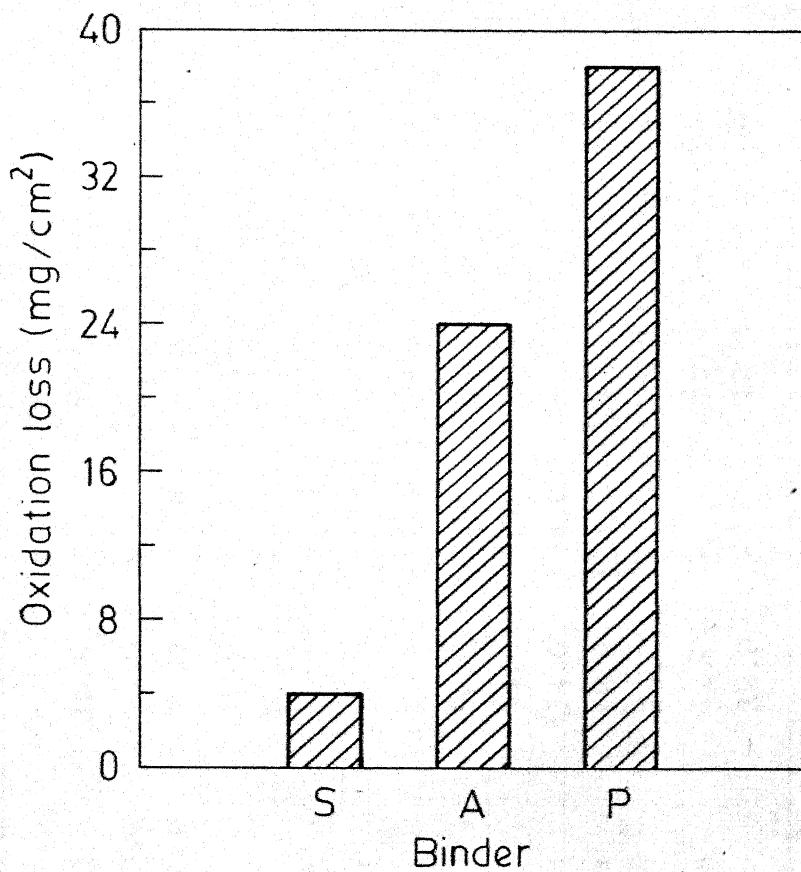


Fig. 1 Effect of binder composition on the oxidation loss of mild steel at 800° C for 5 hrs using coating GC 4.

## COATING CONSTITUTION

Sl. No.	Compo sition Code	A	B	C	Weight D	percentage of components						I
						E	F	G	H	I		
1	2	3	4	5	6	7	8	9	10	11		
1	GC1	65.0	-	15.0	-	-	-	-	-	20.0	-	
2	GC2	30.0	-	10.0	10.0	-	-	50.0	-	-	-	
3	GC3	20.0	10.0	10.0	30.0	-	-	30.0	-	-	-	
4	GC4	20.0	10.0	10.0	30.0	30.0	-	-	-	-	-	
5	GC5	-	25.0	-	15.0	25.0	25.0	10.0	-	-	-	
6	GC6	-	40.0	-	40.0	-	-	-	-	-	20.0	
7	MN1	26.6	10.0	10.0	26.7	26.7	-	-	-	-	-	
8	MN2	12.0	"	"	12.0	56.0	-	-	-	-	-	
9	MN3	56.0	"	"	12.0	12.0	-	-	-	-	-	
10	MN4	12.0	"	"	56.0	12.0	-	-	-	-	-	
11	MN5	34.0	"	"	34.0	12.0	-	-	-	-	-	
12	MN6	12.0	"	"	34.0	34.0	-	-	-	-	-	
13	MN7	34.0	"	"	12.0	34.0	-	-	-	-	-	
14	MG1	26.6	"	"	26.7	-	-	26.7	-	-	-	
15	MG2	12.0	"	"	12.0	-	-	56.0	-	-	-	
16	MG3	56.0	"	"	12.0	-	-	12.0	-	-	-	

Table 3.3 (Contd.)

1	2	3	4	5	6	7	8	9	10	11
17	MG4	12.0	10.0	10.0	56.0	-	12.0	-	-	-
18	MG5	34.0	"	"	34.0	-	12.0	-	-	-
19	MG6	12.0	"	"	34.0	-	34.0	-	-	-
20	MG7	34.0	"	"	12.0	-	34.0	-	-	-
21	CR1	26.6	"	"	26.7	-	-	26.7	-	-
22	CR2	12.0	"	"	12.0	-	-	56.0	-	-
23	CR3	56.0	"	"	12.0	-	-	12.0	-	-
24	CR4	12.0	"	"	56.0	-	-	12.0	-	-
25	CR5	34.0	"	"	34.0	-	-	12.0	-	-
26	CR6	12.0	"	"	34.0	-	-	34.0	-	-
27	CR7	34.0	"	"	12.0	-	-	34.0	-	-

Table 3.4

EFFECT OF COATING COMPOSITIONS USING BINDER 'S'  
OF 1.2 gms/cc SPECIFIC GRAVITY ON THE OXIDATION  
LOSS OF MILD STEEL AT 1000°C FOR 5 hrs\*

Expt. No.	Composition Code	Oxidation Loss mg/cm <sup>2</sup>	Visual Observation	
			After Air drying for 6 hrs	After firing
1	GC1	38.77	B	G
2	GC2	65.60	A	D
3	GC3	24.04	C	D
4	GC4	28.60	A	D
5	GC5	24.94	B	G
6	GC6	43.00	B	D
7	MN1	8.01	C	D
8	MN2	22.93	C	E
9	MN3	23.52	C	E
10	MN4	10.23	C	D
11	MN5	33.97	A	E
12	MN6	44.81	B	E
13	MN7	28.59	C	E
14	MG1	55.86	B	F
15	MG2	86.37	B	G
16	MG3	49.11	B	E
17	MG4	32.95	B	E
18	MG5	40.98	B	F
19	MG6	23.80	B	E
20	MG7	33.72	B	E

Table 3.4 (Contd.)

Expt. No.	Composition Code	Oxidation Loss mg/cm <sup>2</sup>	Visual Observation	
			After Air drying for 6 hrs	After firing
21	CR1	23.71	B	E
22	CR2	22.97	B	E
23	CR3	25.03	B	E
24	CR4	34.48	B	E
25	CR5	60.83	B	F
26	CR6	43.36	B	E
27	CR7	54.24	B	F

\* - 12 cc of the binder was mixed with 20 gms of Coating Powder.

- After firing at high temperature the coating came off in all cases from the specimen surface during cooling.
- Visual observation code.

A - Uneven surface, heavily cracked indicating that the coating has poor paintability and poor adherence to the base.

B - Even surface, slightly cracked at the edges indicating that the coating has moderate paintability and adherence to the base.

C - Even surface, no cracks indicating good paintability and strong adherence to the base.

D - Slight oxidation without pitting.

E - Light gray colour, slightly pits are formed.

F - Dark black coloured oxidation layer which comes off as flakes.

G - Dark gray colour, heavily pitted.

GC 3, the oxidation loss of mild steel at all temperatures decreased rapidly on increasing the specific gravity of the binder upto 1.3 gm/c.c. Beyond this value there was practically no effect at all temperatures. (Figure 2, Table 3.5). Further experimental work was therefore carried out using binder 'S' having a specific gravity of 1.3 gm/c.c.

Several other coating compositions were tried in an attempt to further reduce the oxidation loss. It was observed that using coating compositions MN1 and MN4 the oxidation loss was only 8 and 10 mg/cm<sup>2</sup> (Table 3.4). It was therefore, decided to evaluate these two coating compositions on a variety of ferrous materials at different temperatures and times.

### 3.3 OXIDATION PREVENTION OF MILD STEEL

Effectiveness of coating compositions MN1 and MN4 using binder 'S' of 1.3 gm/cc specific gravity in preventing oxidation loss of mild steel at different temperatures for different times was determined.

Comparing oxidation loss of uncoated mild steel (Figure 3, Table 3.6) with mild steel coated with coating compositions MN1 and MN4 (Figure 4, Table 3.6) and percentage reduction in oxidation loss using these coatings MN1 and MN4 (Figure 5, Table 3.7) following could be observed:

Table 3.5

EFFECT OF SPECIFIC GRAVITY OF THE BINDER 'S' ON  
OXIDATION LOSS OF MILD STEEL IN 5 HRS AT DIFFERENT  
TEMPERATURES USING COATING GC3\*

Expt. No.	Firing Tempe- rature °C	Oxidation Loss (mg/cm <sup>2</sup> )					
		Binder Specific Gravity	and gms/cc	1.1	1.2	1.3	1.4
1	1000	33.70	22.00	10.76	15.55		
2	900	22.79	21.20	9.99	6.21		
3	800	21.94	16.95	5.30	3.47		

\* - 12 cc of the binder was mixed with 20 gms of coating powder.

- In all cases the coated specimens were air dried for 6 hrs. and charged in the furnace at room temperature.
- In all cases the coating surface after air drying was even with no cracks indicating good paintability and adequate adherence to the base.
- After firing at high temperature the coating came off in all cases from the specimen surface during cooling.

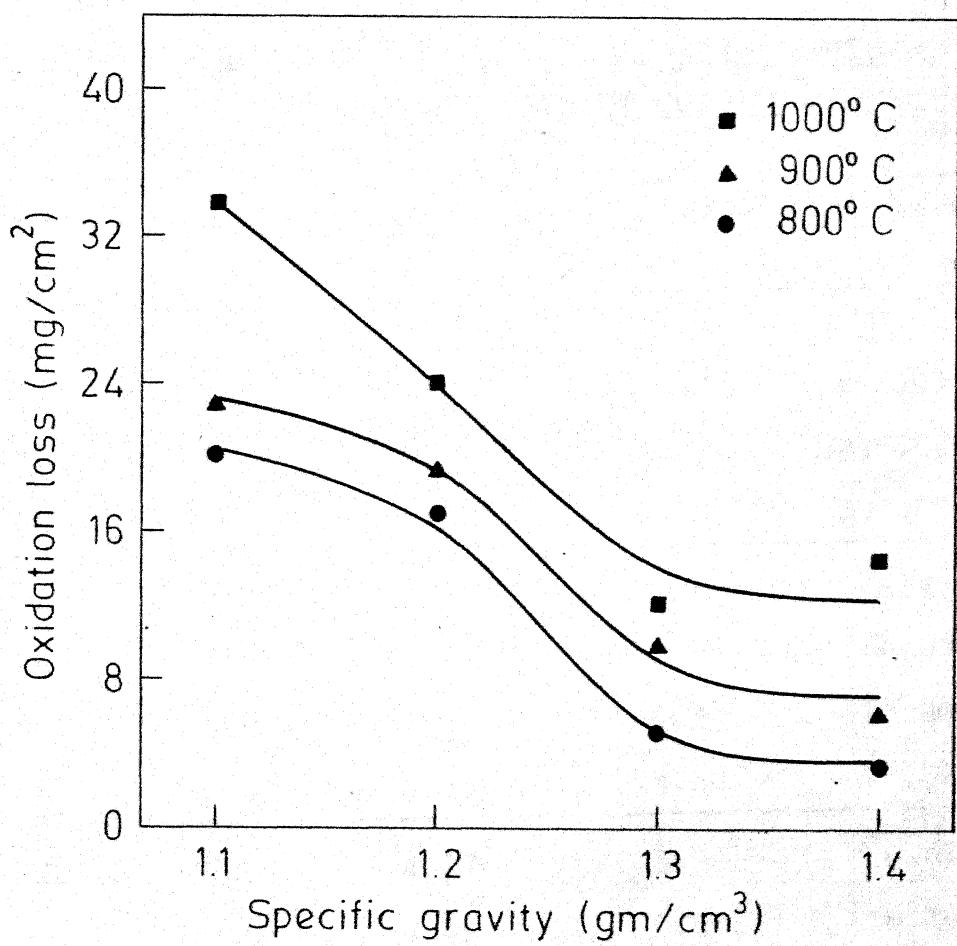


Fig. 2 Effect of specific gravity of the binder on the oxidation loss of mild steel exposed at different temperatures for 5 hours.

Table 3.6OXIDATION LOSS OF MILD STEEL AT DIFFERENT TEMPERATURES AND TIMES WITH AND WITHOUT COATING \*

Expt. No.	Coating Code	Time hrs	Oxidation Loss (mg/cm <sup>2</sup> ) at temperature		
			700°C	800°C	1000°C
1	Uncoated	2.5	6.00	41.35	97.00
2	"	5.0	14.55	54.36	151.90
3	"	7.5	28.98	88.05	298.86
4	"	10.0	46.24	105.28	398.57
5	MN1	2.5	1.79	1.75	8.47
6	"	5.0	3.25	2.01	9.97
7	"	7.5	2.87	6.39	19.80
8	"	10.0	8.53	4.89	22.52
9	MN4	2.5	4.17	5.50	8.61
10	"	5.0	4.45	8.67	18.19
11	"	7.5	3.95	5.02	10.14
12	"	10.0	4.68	5.68	12.84

\* - 12 cc of binder 'S' having a specific gravity of 1.3 gm/cc was mixed with 20 gms of coating powder.

- Coated specimens were air dried for 6 hrs at room temperature before exposure at high temperature.

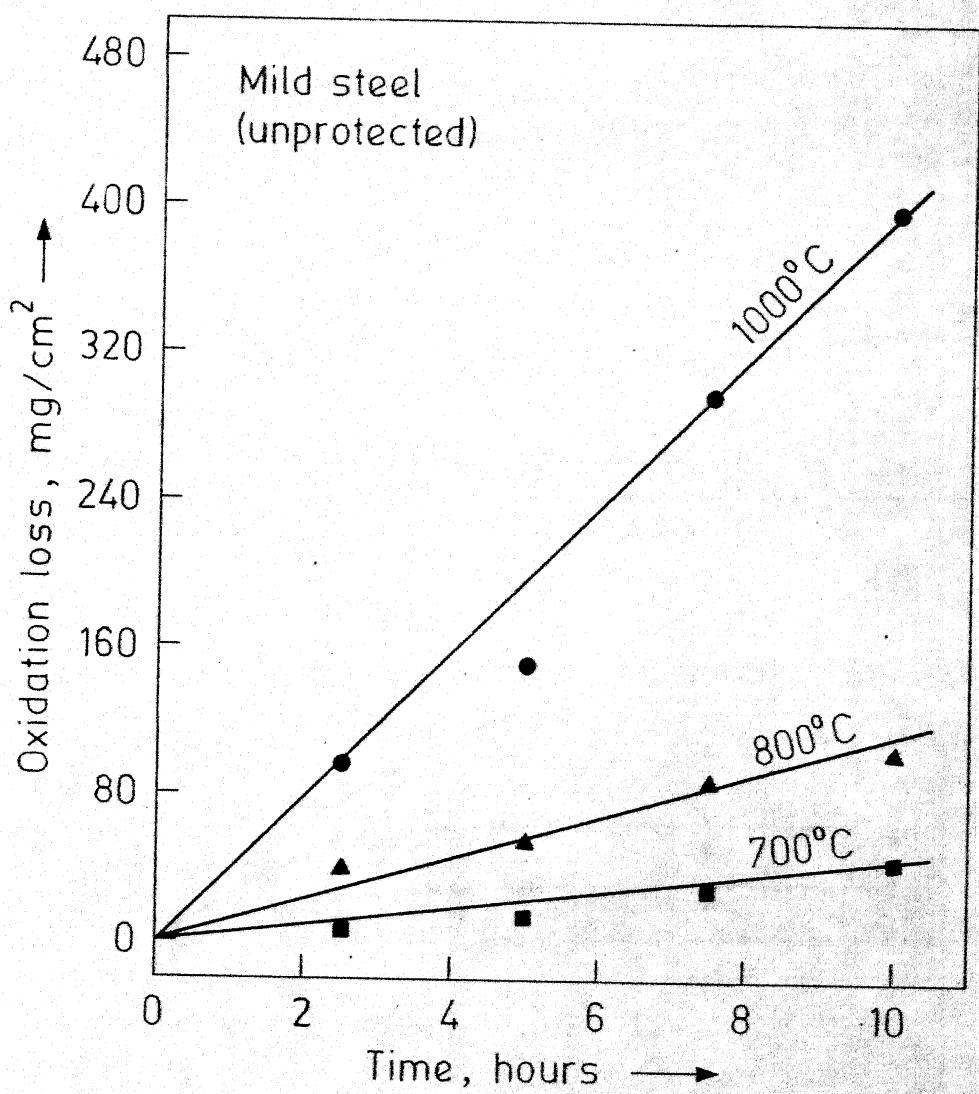


Fig. 3 Oxidation loss of unprotected mild steel at different temperatures and times.

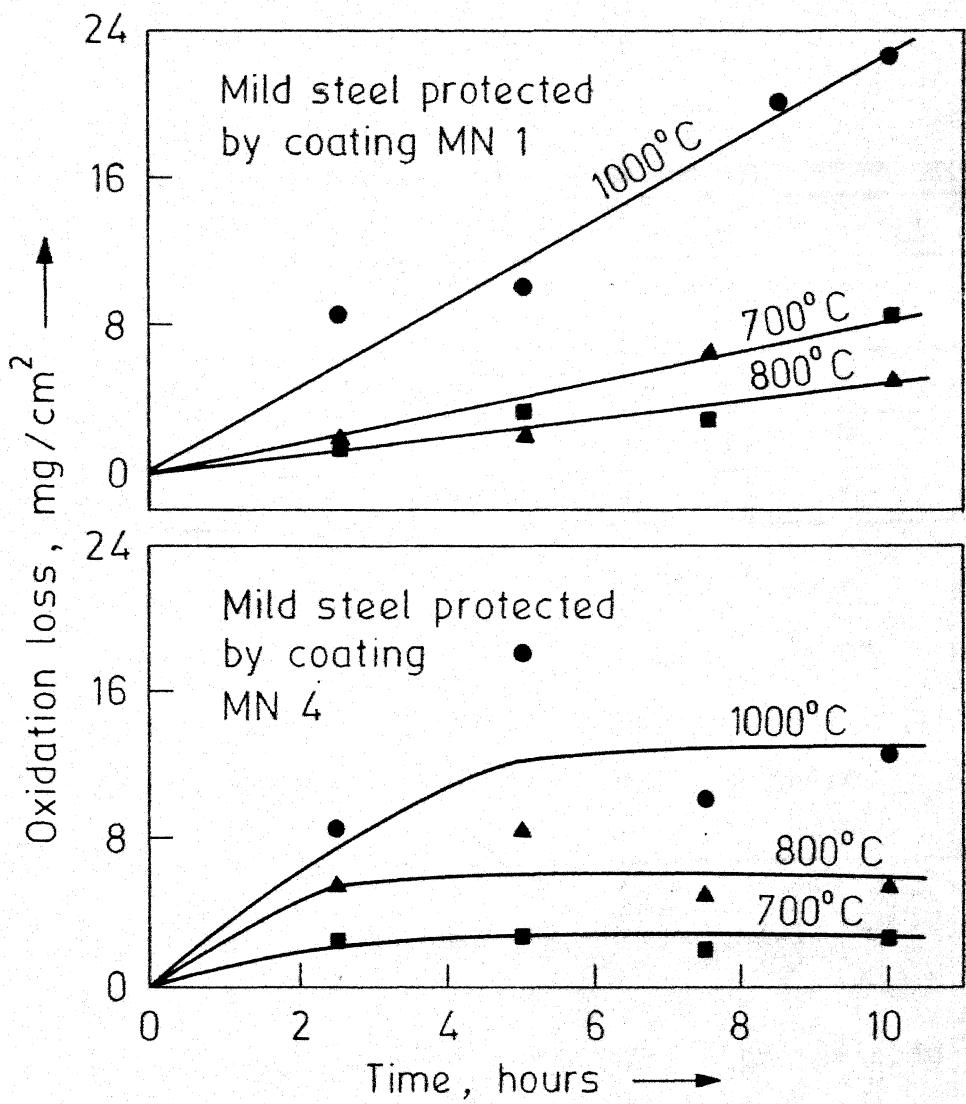


Fig. 4 Oxidation loss of mild steel protected by coatings MN 1 and MN 4 at different temperatures and times.

Table 3.7

PERCENTAGE REDUCTION IN OXIDATION LOSS OF MILD STEEL EXPOSED  
AT DIFFERENT TEMPERATURES AND TIMES USING COATINGS MN1 & MN4\*

Time Hrs.	% Reduction in Oxidation Loss of Mild Steel Using					
	Coating MN1 at			Coating MN4 at		
	700°C	800°C	1000°C	700°C	800°C	1000°C
2.5	70.17	95.77	91.27	30.50	86.70	91.12
5.0	77.66	96.30	93.44	69.42	84.03	88.03
7.5	90.10	92.74	93.37	86.37	94.30	96.61
10.0	81.55	95.36	94.34	89.88	94.80	99.69

\* - 12.cc. of binder S having a specific gravity of 1.3gm/cc.  
was mixed with 20 gms of coating powder.

- Coated specimens were air dried for 6 hrs at room  
temperature before exposure at high temperature.

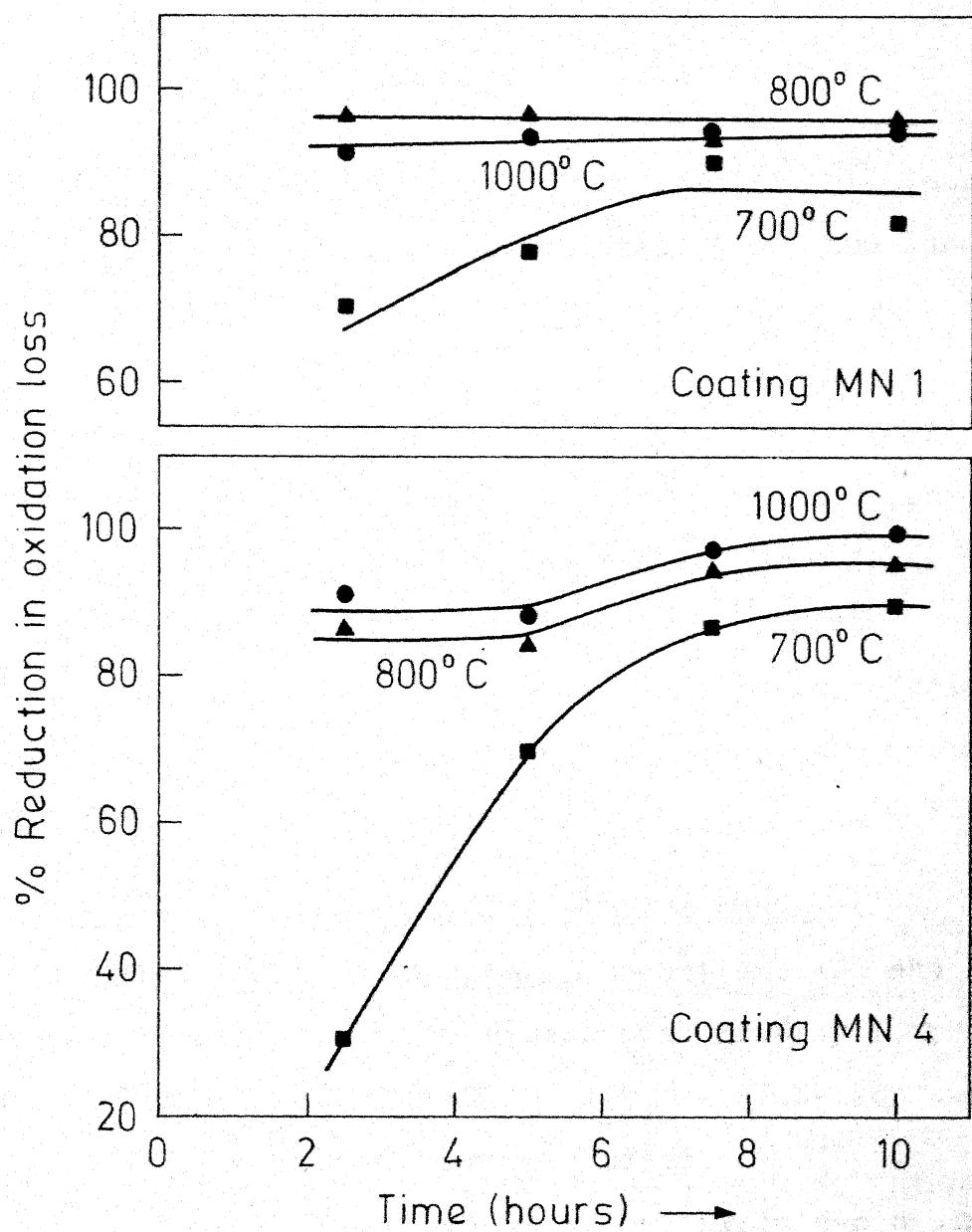


Fig. 5 Effectiveness of coatings MN 1 and MN 4 in reducing oxidation loss of mild steel at different temperatures and times.

- i) Both coatings MN1 and MN4 were very effective in reducing oxidation loss of mild steel at all temperatures and times. At higher temperatures and longer times the coatings are effective in reducing oxidation loss by above 95 %.
- ii) Effectiveness of the coatings in preventing oxidation loss, increases with exposure time at all temperatures, the effect being more pronounced at 700°C.
- iii) At lower temperatures, say 700°C, for shorter exposure periods coating MN1 was more effective in reducing oxidation loss, whereas for longer exposure periods coating MN4 was more effective.

### 3.4 OXIDATION PREVENTION OF STAINLESS STEEL

Effectiveness of coating compositions MN1 and MN4 using binder 'S' of 1.3 gm/c.c. specific gravity in preventing oxidation loss of 18-8 stainless steel at different temperatures for different times was determined.

Comparing oxidation loss of uncoated 18-8 stainless steel (Figure 6, Table 3.8) with specimens protected by coatings MN1 and MN4 (Figure 7, Table 3.8) and percentage reduction in oxidation loss using these coatings (Figure 8, Table 3.9) following can be observed:

Table 3.8

## STEEL

OXIDATION LOSS OF STAINLESS AT DIFFERENT TEMPERATURES  
AND TIMES WITH AND WITHOUT COATING\*

Expt. No.	Composi- tion Code	Time hrs.	Oxidation Loss (mg/cm <sup>2</sup> ) at temperature			
			900°C	1000°C	1100°C	1200°C
1	Uncoated	2.5	5.74	18.65	48.90	76.00
2	"	5.0	9.13	24.90	43.88	111.94
3	"	7.5	22.06	22.45	68.42	152.50
4	"	10.0	23.03	34.25	88.00	160.94
5	MN1	2.5	0.00	0.04	1.46	3.43
6	"	5.0	0.00	0.11	1.83	3.38
7	"	7.5	0.00	0.34	2.83	11.20
8	"	10.0	0.00	0.55	6.45	16.52
9	MN4	2.5	0.00	0.21	1.43	6.23
10	"	5.0	0.00	1.92	1.96	11.42
11	"	7.5	0.00	1.99	2.31	10.00
12	"	10.0	0.00	3.76	2.75	12.25

\* - 12 cc of binder S having a specific gravity of 1.3 gm/cc was mixed with 20 gms of coating powder.

- Coated specimens were air dried for 6 hrs at room temperature before exposure at high temperature.

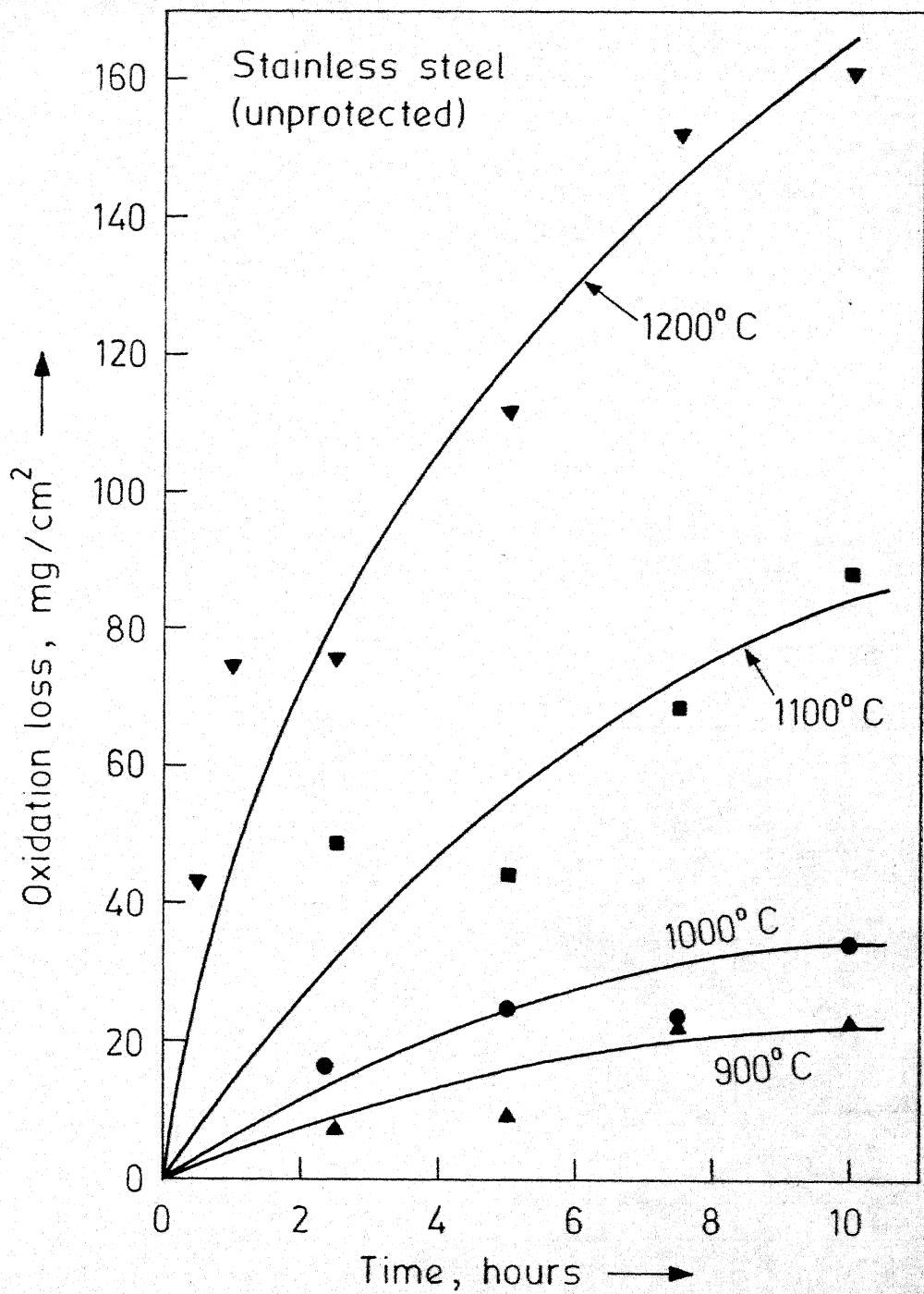


Fig. 6 Oxidation loss of unprotected stainless steel at different temperatures and times.

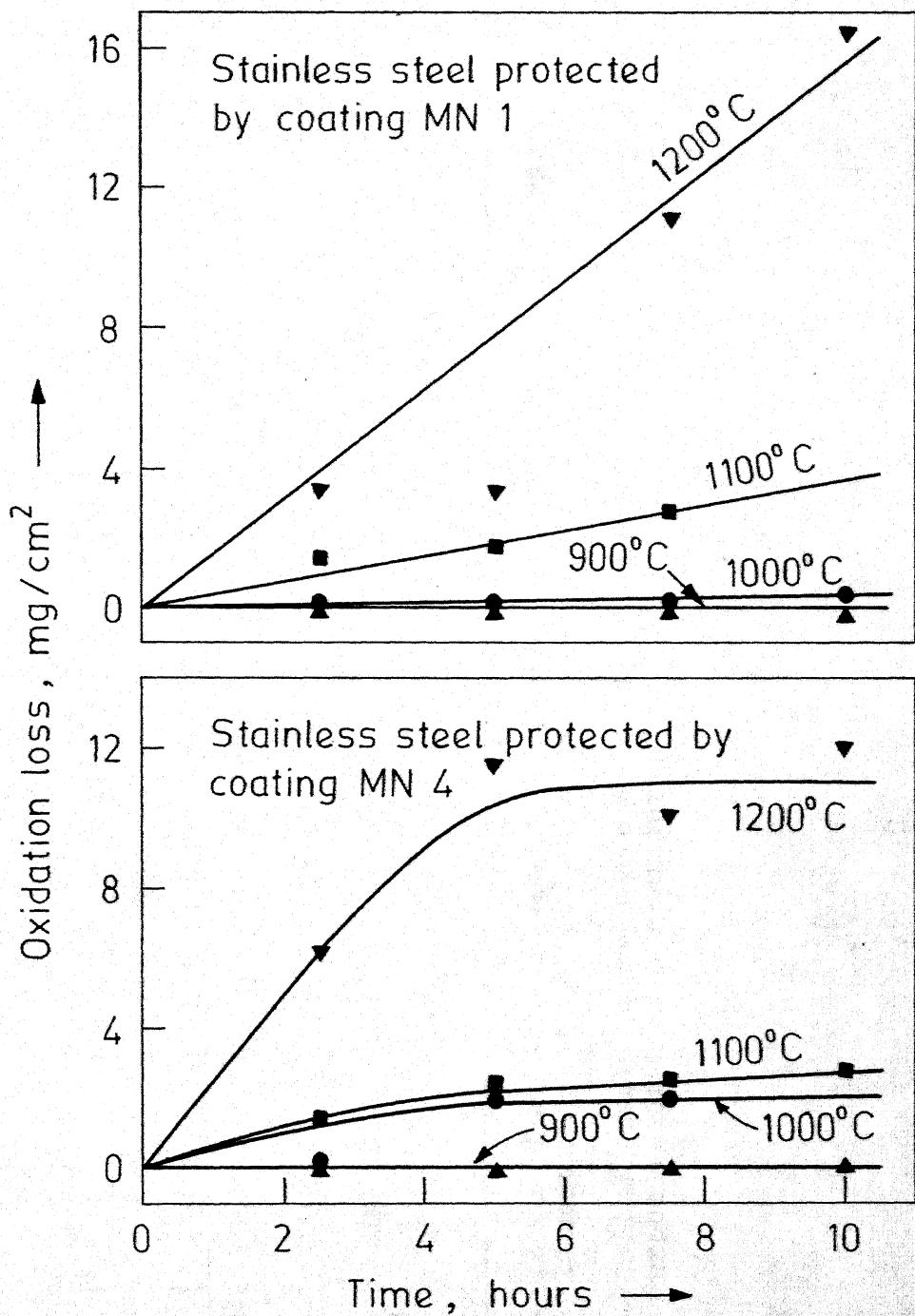


Fig. 7 Oxidation loss of stainless steel protected by coatings MN 1 and MN 4 at different temperatures and times.

Table 3.2

PERCENTAGE REDUCTION IN OXIDATION LOSS OF 18-8 STAINLESS STEEL  
EXPOSED AT DIFFERENT TEMPERATURES AND TIMES USING COATINGS  
MNL AND MN4\*

Time hrs.	Percentage Reduction in oxidation loss of stainless steel using				
	Coating MNL at 900°C	Coating MNL at 1000°C	Coating MNL at 1100°C	Coating MN4 at 900°C	Coating MN4 at 1000°C
2.5	100.00	99.81	97.02	95.49	100.00
5.0	100.00	99.49	95.55	96.48	100.00
7.5	100.00	98.49	95.56	92.66	100.00
10.0	100.00	98.41	92.67	89.74	100.00

\* - 12 cc of binder 'S' having a specific gravity of 1.3 gm/cc was mixed with 20 gms of coating powder.

- Coated specimens were air dried for 6 hrs at room temperature before exposure at high temperature.

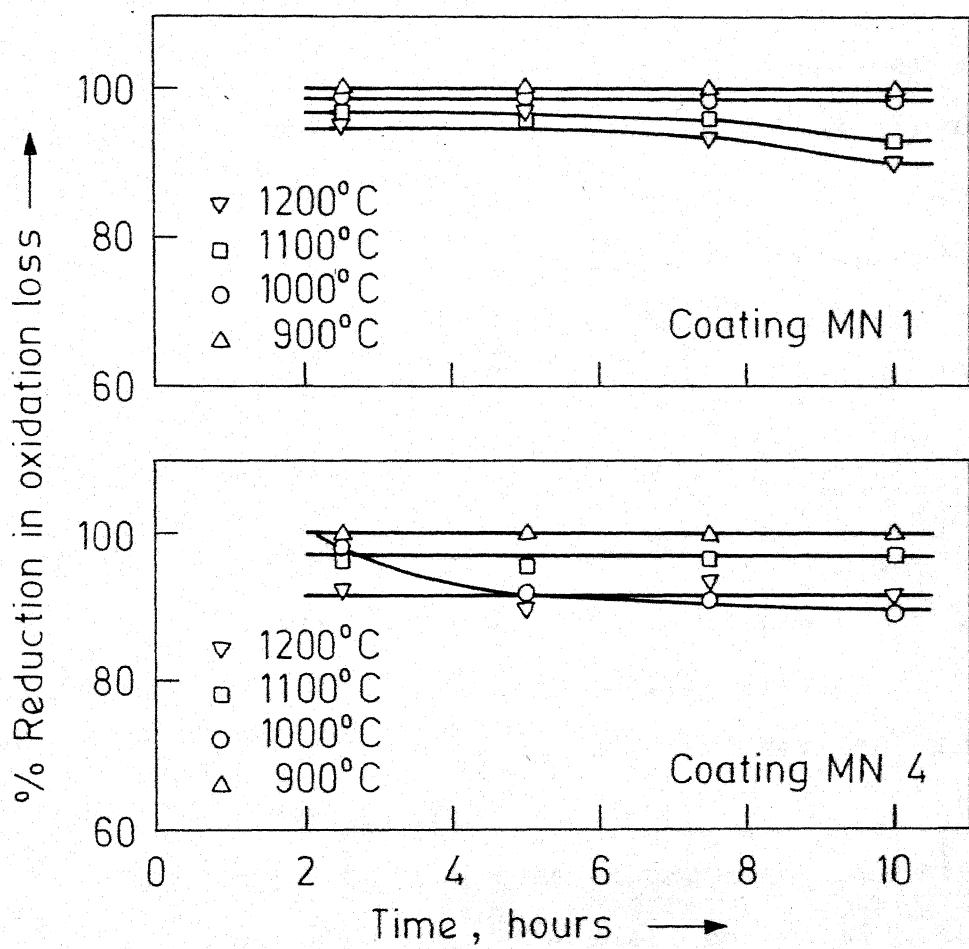


Fig. 8 Effect of coatings MN 1 and MN 4 in reducing oxidation loss of tool steel at different temperatures and times.

- i) Both coatings MN1 and MN4 drastically reduced the oxidation loss of 18-8 stainless steel at all temperatures and times.
- ii) It appeared that at all temperatures and times there was practically no difference in both the coatings, percentage reduction in oxidation loss being above 89 % in all cases.

### 3.5 OXIDATION PREVENTION OF TOOL STEEL

Effectiveness of coating compositions MN1 and MN4 using binder 'S' of 1.3 gm/c.c. specific gravity in preventing oxidation loss of tool steel at different temperatures for different times was determined.

Comparing oxidation loss of uncoated tool steel (Figure 9, Table 3.10) with tool steel coated with coating compositions MN1 and MN4 (Figure 10, Table 3.10) and percentage reduction in oxidation loss using these coatings (Figure 11, Table 3.11) following could be observed:

- i) Coating - MN4 was very effective in reducing oxidation loss of tool steel at all temperatures and times, coating MN1 was effective at  $900^{\circ}\text{C}$  and  $1000^{\circ}\text{C}$  for all times. However at  $1100^{\circ}\text{C}$  its effectiveness decreased with increasing time.

Table 3.10

OXIDATION LOSS OF TOOL STEEL AT DIFFERENT TEMPERATURES  
AND TIME WITH AND WITHOUT COATING\*

Expt. No.	Composition Crde	Time hrs.	Oxidation loss mg/cm <sup>2</sup> at Exposure Temperature		
			900°C	1000°C	1100°C
1	Uncoated	1.0	8.50	28.25	247.85
2	"	2.0	29.94	102.64	280.64
3	"	3.0	28.99	294.41	416.64
4	"	4.0	94.76	318.91	426.25
5	"	5.0	102.53	339.30	465.23
6	MN1	1.0	3.25	10.98	28.28
7	"	2.0	8.97	14.86	50.24
8	"	3.0	7.26	15.32	101.00
9	"	4.0	10.23	16.21	163.89
10	"	5.0	11.36	19.71	327.23
11	MN4	1.0	3.98	15.56	13.08
12	"	2.0	7.42	18.42	17.06
13	"	3.0	9.29	20.06	21.89
14	"	4.0	9.64	22.45	32.05
15	"	5.0	9.89	23.93	85.98

\* - 12 cc of binder S having a specific gravity of 1.3 gm/cc was mixed with 20 gms of coating powder.

- Coated specimens were air dried for 6 hrs at room temperature before exposure at high temperature.

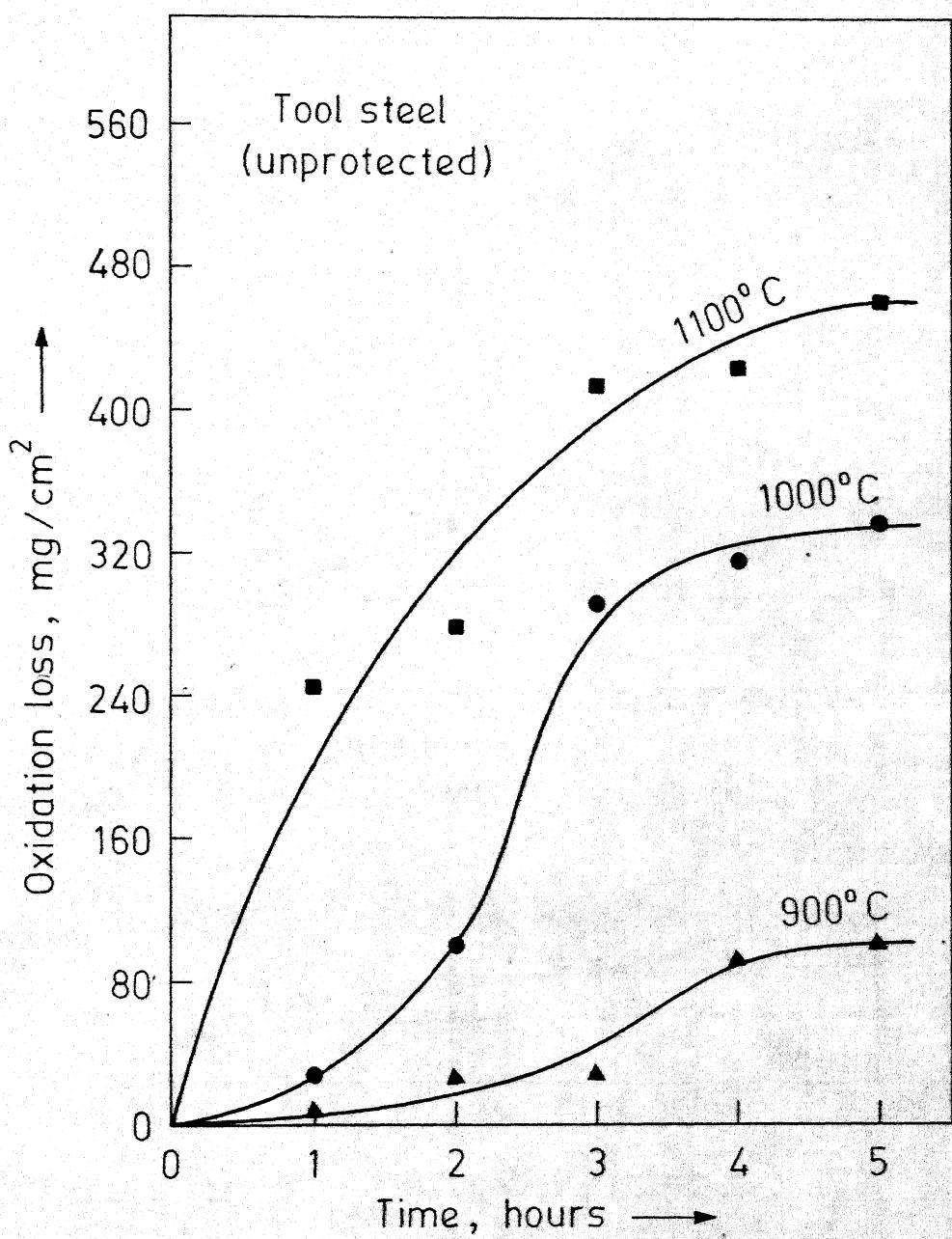


Fig. 9 Oxidation loss of unprotected tool steel at different temperatures and times.

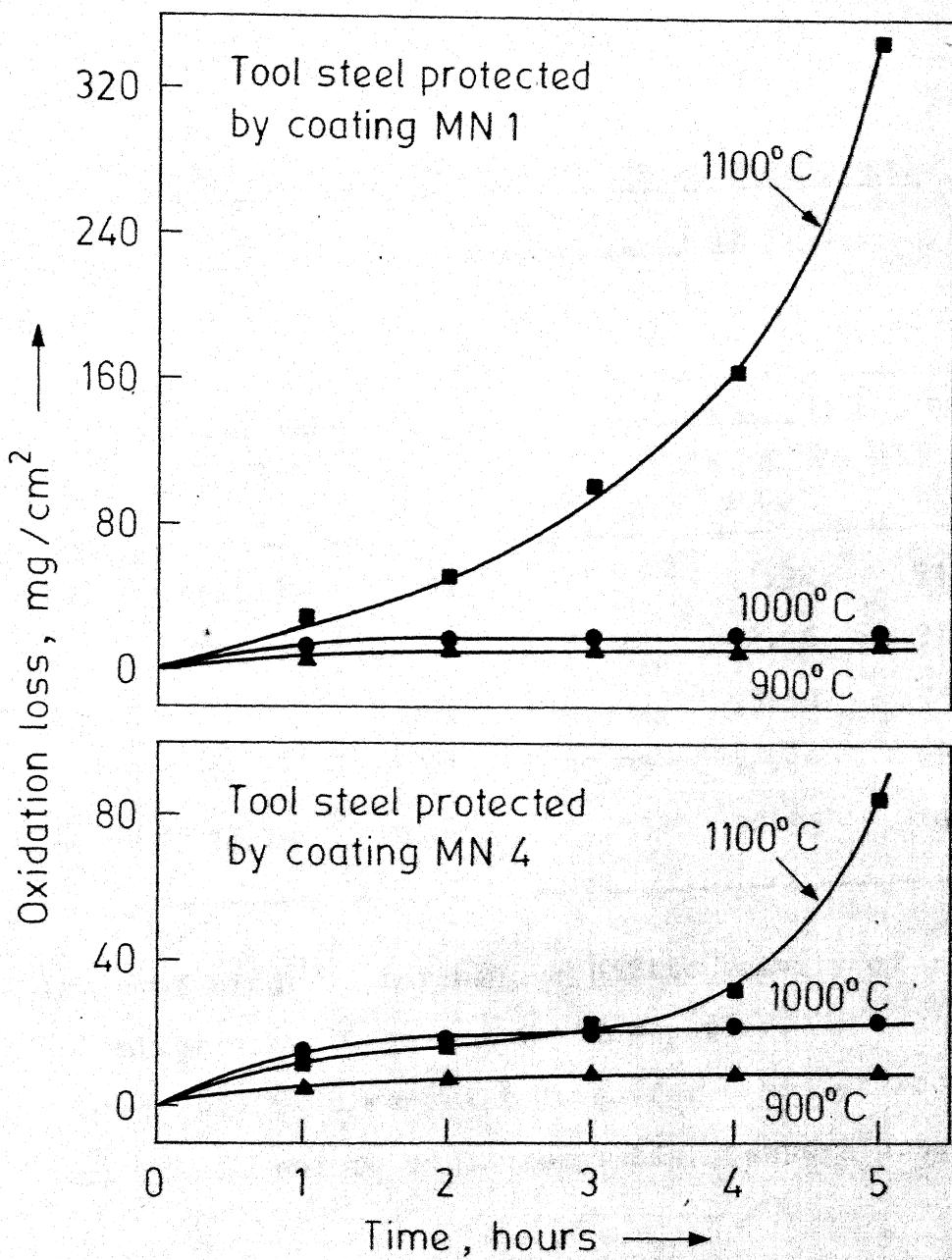


Fig. 10 Oxidation loss of tool steel protected by coatings MN 1 and MN 4 at different temperatures and times.

Table 3.11

PERCENTAGE REDUCTION IN OXIDATION LOSS OF TOOL STEEL  
EXPOSED AT DIFFERENT TEMPERATURES AND TIMES USING  
COATINGS MN1 AND MN4 \*

Time hrs.	Percentage Reduction in oxidation loss of Tool Steel Using						
	Coating MN1 at			Coating MN4 at			
	900°C	1000°C	1100°C	900°C	1000°C	1100°C	
1.0	61.76	61.13	88.59	53.18	44.92	94.72	
2.0	70.04	85.52	82.10	75.22	82.05	93.92	
3.0	74.96	94.80	75.76	67.95	93.19	94.75	
4.0	89.70	94.91	61.55	89.83	92.96	92.48	
5.0	88.92	94.19	29.66	90.35	92.95	81.52	

\* - 12 cc of binder S having a specific gravity of 1.3 gm/cc  
 was mixed with 20 gms of coating powder.  
 - Coated specimens were air dried for 6 hrs at room  
 temperature before exposure at high temperature.

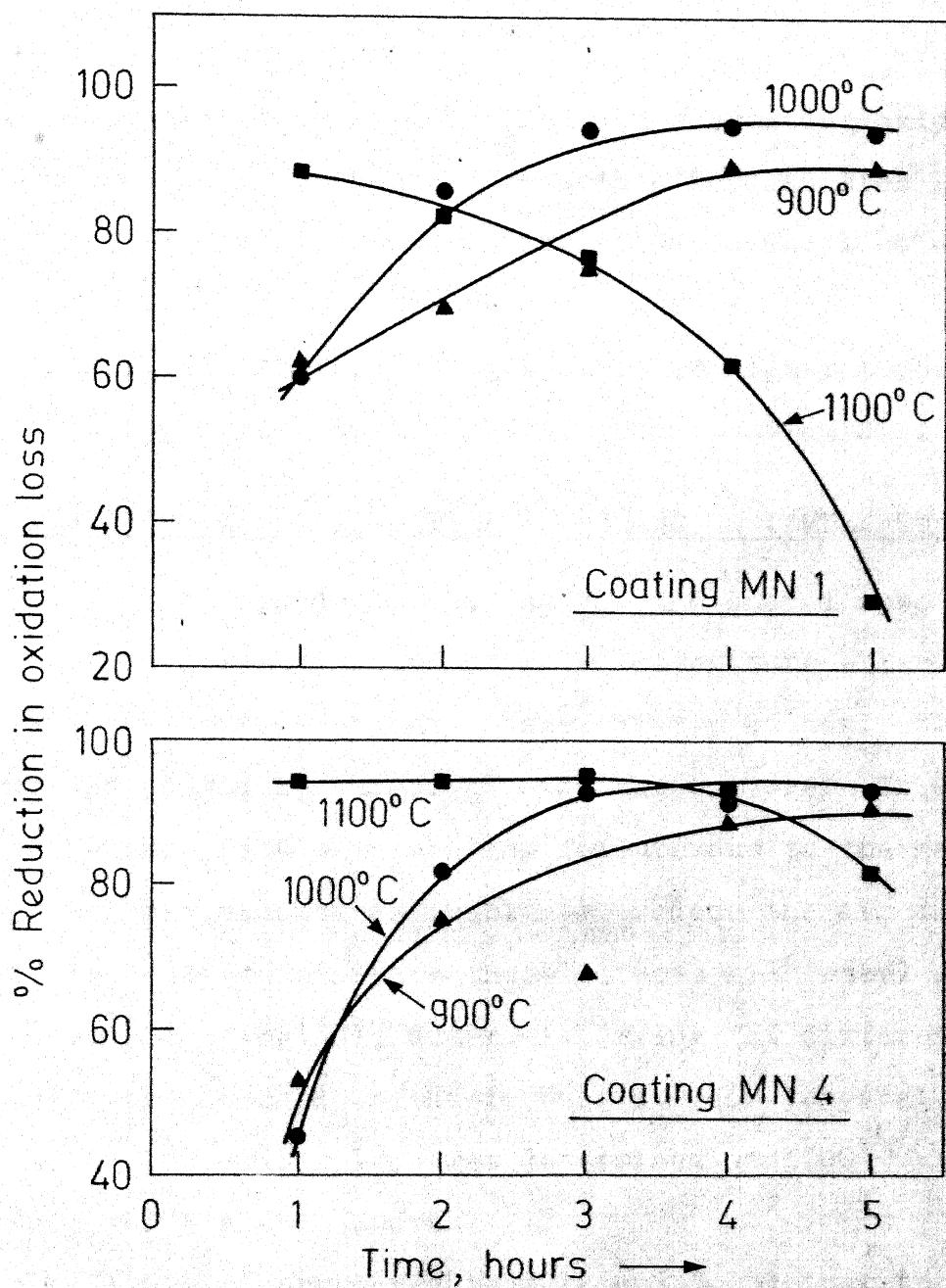


Fig. 11 Effectiveness of coatings MN 1 and MN 4 in reducing oxidation loss of tool steel at different temperatures and times.

- ii) Effectiveness of the coatings in preventing oxidation, as judged by percentage reduction in oxidation loss increases with exposure time at  $900^{\circ}\text{C}$  and  $1000^{\circ}\text{C}$ . At  $1100^{\circ}\text{C}$  it decreased.
- iii) It appears that for longer times at higher temperature coating MN4 is more effective than coating MN1.

### 3.6 EFFECT OF AIR DRYING TIME AND CHARGING TEMPERATURE

In the work described, so far the coated specimens were charged in the furnace at room temperature after air drying for 6 hours. In many cases, it may be desirable to charge the coated specimens at high temperature. This could however, result in coating failure due to thermal shock. It may also be desirable to reduce the air drying time. In order to evaluate these effects mild steel specimens coated with coating MN1, after air drying for different periods, were charged in the furnace at room temperature and at  $1000^{\circ}\text{C}$ . Oxidation loss was determined at  $1000^{\circ}\text{C}$  after exposure for 5 and 10 hours.

It was observed (Table 3.12 and 3.13) that the oxidation loss of mild steel decreases with increasing air drying time on charing at either  $1000^{\circ}\text{C}$  or at room temperature. As the air drying time is increasing more and more water from the coating gets evaporated which in turn reduces the possibility

Table 3.12

EFFECT OF AIR DRYING TIME AND CHARGING TEMPERATURE  
ON THE OXIDATION LOSS OF MILD STEEL AT 1000°C.\*

Coating Code	Charging Temp.	Oxidation Loss $\text{mg/cm}^2$	
		Exposed at 1000°C for 5 hrs	Exposed at 1000°C for 10 hrs
	Air drying time hrs.	Air drying time hrs.	
	1	2	4
	6	1	6
MN1	1000°C	19.72	16.70
		10.95	7.30
		47.02	74.57
		68.11	12.25
II	Room Temp.	108.69	21.39
		77.05	9.90
		76.15	35.26
		64.73	22.52

\* - 12 cc of Binder '3' having a specific gravity of 1.3 gm/cc was mixed with 20 gms of coating powder.

Table 3.13

PERCENTAGE REDUCTION IN OXIDATION LOSS OF MILD STEEL AIR  
DRIED FOR DIFFERENT PERIODS AND EXPOSED AT 1000°C FOR

5 Hrs and 10 Hrs \*

Coating Code	Charging Temp.	Percentage Reduction in Oxidation Loss		Exposed at 1000°C for 5 hrs	Exposed at 1000°C for 10 hrs
		Air drying time hrs	Air drying time hrs		
		1	2	4	6
MNL	1000°C	87.02	89.01	92.18	95.20
"	Room Temp.	28.32	85.92	49.28	93.49

\* - 12 cc of Binder 'S' having a specific gravity of 1.3 gm/cc was mixed  
with 20 gms of coating powder.

of coating damage during firing because of sudden moisture evaporation. This would explain the benificial effect of air drying for longer times. Although the oxidation loss after air drying for 1 hour was more than the loss after air drying for 6 hours, however, it still represented a reduction in oxidation loss of 87% for 5 hrs exposure and 88% for 10 hrs at  $1000^{\circ}\text{C}$  as compared to uncoated specimens. This may be more than adequate for many industrial applications.

Charging at  $1000^{\circ}\text{C}$  showed less oxidation loss in most of the cases. It may be because, while charging at room temperature the poracity of the coating is more, till the coating becomes viscous enough to fill the pores. But in case of charing at  $1000^{\circ}\text{C}$  the coating becomes viscous and fills the pore immediately on charging, thereby reducing the time for which the specimens remain practically unprotected. This results in lower oxidation loss in cases when the specimens are charged in the furnace at  $1000^{\circ}\text{C}$  as compared to charging in furnace at room temperature and raising the temperature afterwards.

### 3.7 THERMAL CONDUCTIVITY OF THE COATING

Ideal coating should have good thermal conductivity. Since, poor conductivity would mean that the time taken by the coated specimen to attain the furnace temperature would be different than the uncoated specimen. Therefore, adjustments

will have to be made in the normal heat treatment cycle. However, if the thermal conductivity of the coating is good, then, such coating could be used without altering the conventional heat treatment cycle.

To evaluate the thermal conductivity of the coating and the time taken to attain the furnace temperature at the centre of mild steel specimen, 2.5 cm (dia) x 5 cm (long), in coated and uncoated conditions.

Three specimens, one without coating and the other two with coating MN1 and MN4 were charged in the furnace at room temperature and the temperature has been raised. The temperatures of the specimens were measured after 5 minutes interval. The results clearly show that there was practically no difference in temperature of coated and uncoated specimens indicating both the coatings have good thermal conductivity (Figure 12).

### 3.8 EFFECT OF COATING ON THE HARDENING TREATMENT FOR STEELS

As stated earlier, an ideal coating should not hinder the normal heat treatment process so that it could be used without altering the conventional heat treatment processes.

To evaluate the effect of coating on material hardenability two specimens one without coating and the other with coating MN1 were austenitized at  $1000^{\circ}\text{C}$  for 1 hr followed by water quenching. Microhardness at 0.127 mm interval was

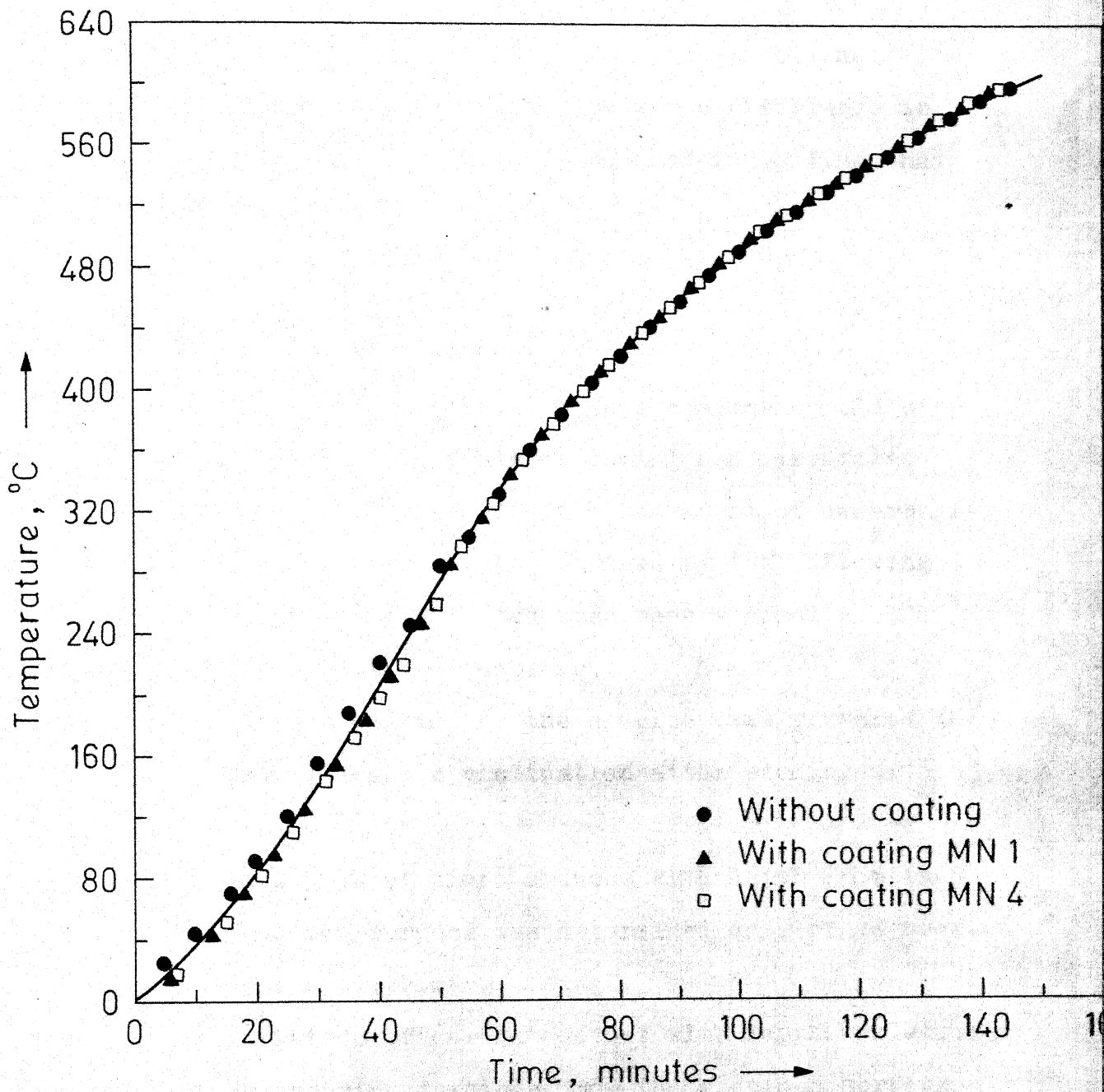


Fig. 12 Rise in temperature at the centre of 25 mm diameter mild steel specimen without coating and with coatings MN 1 and MN 4.

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measured on a cross section of each of the specimens. The results show that there was practically no difference in hardness of coated and uncoated specimens indicating that the coating is not hindering the hardening treatment (Figure 13, Table 3.14).

### 3.9 PREVENTION OF DECARBURIZATION

As mentioned earlier, an ideal coating should also prevent decarburization of steels during heat treatment process. To evaluate this effect, the extent of decarburization, in a 0.56% carbon steel studied by the following methods for coated and uncoated specimens exposed at  $1000^{\circ}\text{C}$  for 10 hrs followed by air cooling,

- i) A cross section of the specimen was prepared for metallographic examination after etching with 5% Nital.
- ii) Variation of microhardness with depth from the specimen surface was determined on a cross section of each specimen.
- iii) Variation of carbon content with depth was studied by removing turnings from the specimen surface in steps of 0.127 mm upto a depth of 1.143 mm. These turnings were analysed for carbon content using Strohleim apparatus.

Table 3.14

EFFECT OF COATING ON HARDENING TREATMENT OF A 0.56% C STEEL SPECIMEN AUSTENITISED AT 1000°C FOR 1 HR FOLLOWED BY WATER QUENCHING

Distance from surface mm	Microhardness D.P.N.	
	Uncoated	Coated with Mn1
0.00	575.6	570.00
0.13	570.0	565.0
0.25	565.6	557.5
0.38	552.5	547.5
0.51	537.5	527.5
0.64	516.4	512.5
0.76	441.2	441.2
0.89	401.2	401.2
1.40	376.4	376.4
1.91	344.4	344.4
2.42	344.4	344.4
3.00	344.4	344.4
3.58	344.4	344.4
4.09	344.4	344.4
4.60	376.4	376.4
5.11	401.2	401.2
5.24	441.2	441.2
5.37	516.4	512.5
5.50	537.5	527.5
5.63	552.5	547.5
5.76	565.6	557.5
5.89	570.0	565.0
6.00	575.6	570.0

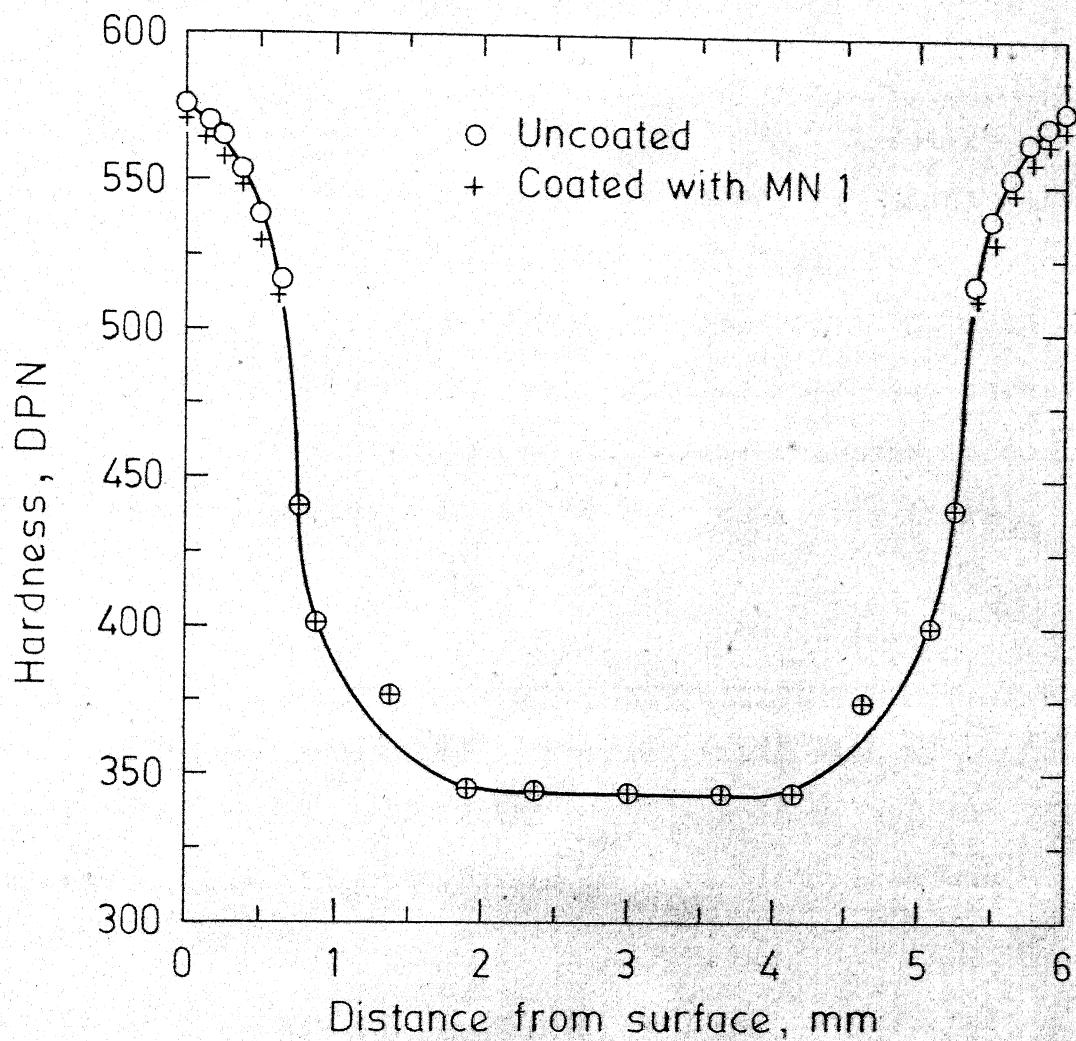


Fig. 13 Effect of coating MN 1 on the hardenability of 0.56 % carbon steel specimen austenitized at  $1000^{\circ}\text{C}$  for 1 hour followed by water quenching.

Taking into consideration the loss in specimen thickness during exposure due to oxidation it could be seen that in coated specimen both hardness and carbon content (Figure 14) attained the value of the parent material in annealed condition at a lesser depth than the uncoated specimen. This indicated that the coating MN1 was helping in preventing decarburization. This fact was also borne out by the photomacrographs of the cross-sections of coated and uncoated specimens after the heat treatment cycle (Figure 15, Table 3.15 and 3.16).

### 3.10 SURFACE COVERING ABILITY OF THE COATING

20 gms of coating powder was mixed with 12 cc of binder and coated on the mild steel specimens and the weight of powder plus binder required to cover 1 m<sup>2</sup> area was calculated as follows.

Weight of the uncoated specimen	$(W_1)$	= 12.3298 gms
Weight of the coated specimen	$(W_2)$	= 16.3021 gms
Specimen surface area	$(A)$	= 50.96 cm <sup>2</sup>
Weight of 12 cc of binder	$(W_3)$	= 15.6 gms
Weight of powder used	$(W_4)$	= 20.0 gms
Weight of powder + binder to cover 50.96 cm <sup>2</sup> surface area		= $W_2 - W_1$ = 3.9723 gms

Table 3.15

VARIATION OF MICROHARDNESS WITH DEPTH IN 0.56% C STEEL SPECIMENS, UNCOATED AND COATED WITH MN1, AFTER HEATING AT 1000°C FOR 10 Hrs FOLLOWED BY AIR COOLING

Uncoated		Coated with MN1	
Distance from surface mm	Micro-hardness D.P.N.	Distance from surface mm	Microhardness D.P.N.
1.07	85.2	0.66	161.0
1.19	110.2	0.79	92.4
1.32	78.0	0.91	137.2
1.45	106.6	1.04	154.4
1.57	161.0	1.1 <del>17</del>	168.0
2.08	168.0	1.68	93.4
2.59	168.0	2.18	148.4
3.10	168.0	2.69	168.0
3.61	168.0	3.26	168.0
4.11	168.0	3.71	168.0

Table 3.16

VARIATION OF CARBON CONTENT WITH DEPTH IN 0.56% C  
STEEL SPECIMENS, UNCOATED AND COATED WITH MN1,  
AFTER HEATING AT 1000°C FOR 10 Hrs FOLLOWED BY  
AIR COOLING

Uncoated		Coated with MN1	
Distance from surface mm	Carbon percentage	Distance from surface mm	Carbon percentage
1.07	0.10	0.66	0.11
1.19	0.08	0.79	0.12
1.32	0.19	0.91	0.15
1.45	0.25	1.04	0.22
1.57	0.33	1.17	0.28
1.70	0.40	1.30	0.29
1.83	0.44	1.42	0.35
1.96	0.43	1.55	0.40
2.08	0.44	1.68	0.51

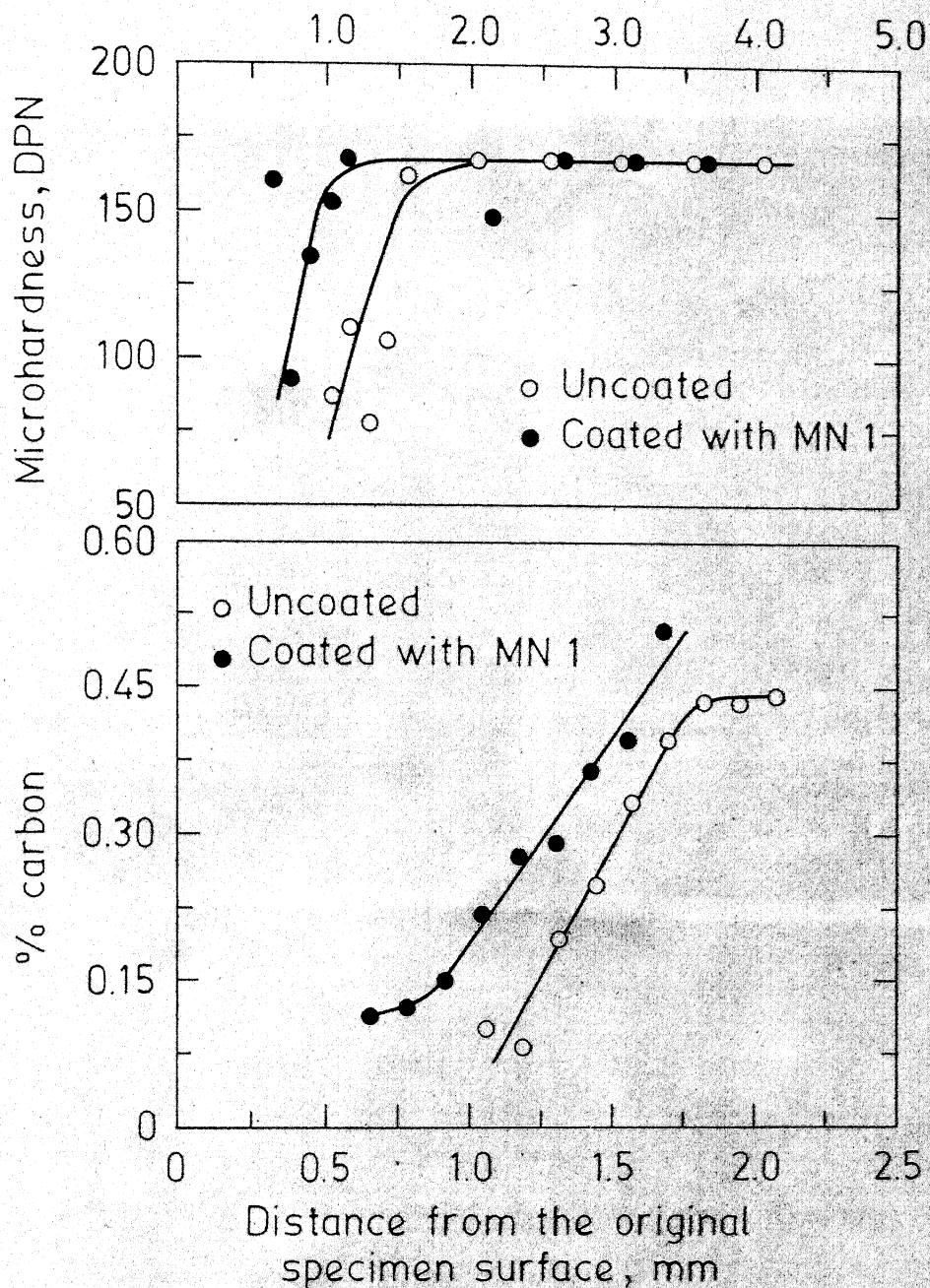


Fig. 14 Variation of microhardness and carbon content with depth in 0.56 % carbon steel specimens, uncoated and coated with MN 1, after heating at 1000°C for 10 hours followed by air cooling.

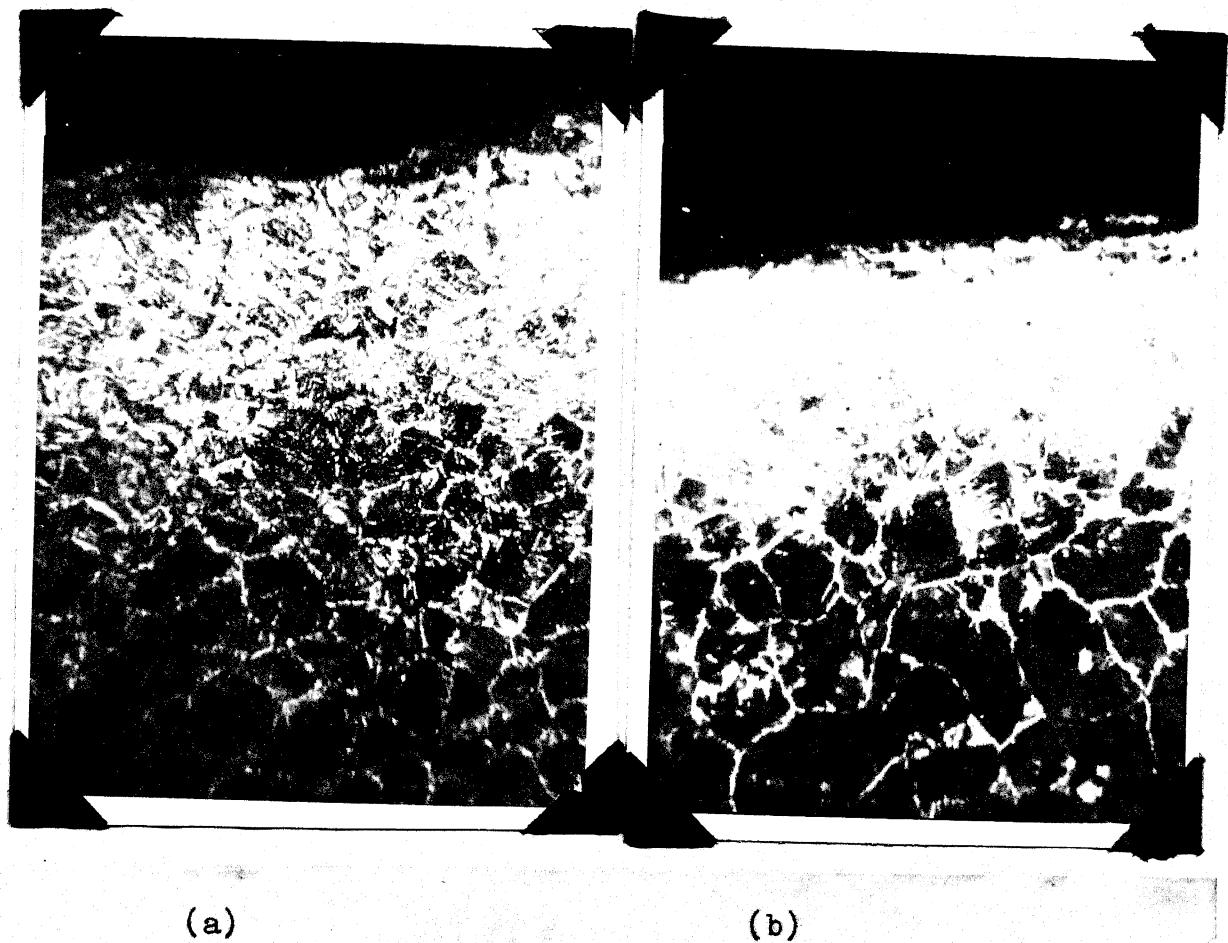


Fig. 15: Microstructures showing the decarburized layers in (a) uncoated and (B) coated with MN1 specimens of 0.56% C steel exposed at 1000°C for 10 hrs. ( 150X).

Weight of powder + binder  
to cover 1 m<sup>2</sup> surface area

$$= \frac{10^4}{A} \times (W_2 - W_1)$$

$$= \frac{10^4 \times 3.9723}{50.96}$$

$$= 779.49 \text{ gms}$$

Weight of powder out of  
779.49 gms of binder + powder

$$= \frac{779.49}{W_3 + W_4} \times W_4$$

$$= \frac{779.49 \times 20.0}{15.6 + 20.0}$$

$$= 437.91 \text{ gms}$$

Weight of binder

$$= 779.49 - 437.91$$

$$= 341.58 \text{ gms}$$

Volume of 341.58 gms of binder  
solution

$$= \frac{341.58}{W_3} \times 12$$

$$= \frac{341.58}{15.6} \times 12$$

$$= 262.75 \text{ cc}$$

To cover 1 m<sup>2</sup> surface area 437.91 gms of powder and  
262.75 cc of binder solution would be required.

Or in other words 20 gms of powder and 12 cc of binder  
solution mixed together would cover about 450 cm<sup>2</sup> surface  
area.

### 3.11 COST OF COATING

The cost of coating has been calculated on the basis of present raw material cost without grinding and labour charges as follows.

#### Raw Materials Cost

Material 'A'	Rs. 500/Ton
Material 'B'	Rs. 2000/Ton
Material 'C'	Rs. 500/Ton
Material 'D'	Rs. 500/Ton
Material 'E'	Rs. 500/Ton
Binder 'S'	Rs. 2000/Ton

To get 1.3 gm/cc specific gravity of binder solution 75 gms of binder was added to 100 cc of water.

Total volume of the binder solution  
i.e. 75 gms of binder + 100 cc of  
water = 110 cc

To cover 1 m<sup>2</sup> surface area volume  
of biner required = 262.75 cc

Weight of the Binder added to get  
262.75 cc of solution =  $\frac{262.75}{110} \times 75$   
= 179.15 gms

The cost of 10 Kg of coating MN1  
powder (in the proportion as  
given in Table 3.3) = Rs. 10.55

The cost of 10 Kg of coating MN4  
powder (in the proportion as  
given in Table 3.3) = Rs. 8.76

The price of 179.15 gms of the binder	$= \text{Rs. } \frac{179.15}{10^5} \times 2000$
	$= \text{Rs. } 0.36$
To cover 1 m <sup>2</sup> surface area required powder	$= 437.91 \text{ gms}$
The price of 437.91 gms coating MN1	$= \text{Rs. } \frac{437.91}{10^4} \times 10.55$
	$= \text{Rs. } 0.46$
Similarly the price of 437.91 gms of coating MN4	$= \text{Rs. } 0.38$
The price of coating MN1 powder + binder 'S' to cover 1 m <sup>2</sup> surface area	$= \text{Rs. } (0.46 + 0.36)$
	$= \text{Rs. } 0.82$
Similarly the price of coating MN4 powder + binder 'S' to cover 1 m <sup>2</sup> surface area	$= \text{Rs. } (0.38 + 0.36)$
	$= \text{Rs. } 0.74$

### 3.12 REMOVAL OF THE COATING AFTER EXPOSURE

The surface coatings for heat treatment process ideally should have the properties stated in section 1.3. Two important properties are it should come off by itself from the specimen surface and it should not react with the specimen surface when exposed at high temperatures.

It was observed (Table 3.17) that both coatings MN1 and MN4 came off by themselves during cooling of the specimen after exposure at high temperature and also they are not

TABLE 3/17 SURFACE APPEARANCE OF UNPROTECTED AND PROTECTED SPECIMENS  
AFTER EXPOSURE AT DIFFERENT TEMPERATURES AND TIMES.

Coating code	Exposure time hrs	MILD STEEL				STAINLESS STEEL				TOOL STEEL			
		Exposure temperature °C		Exposure time hrs		Exposure temperature °C		Exposure time hrs		Exposure temperature °C		Exposure time hrs	
		700	900	1100	1300	900	1100	1300	1500	900	1100	1300	1500
MIL (Untreated specimen)	2.5	S	E	2.5	E	E	E	E	E	E	E	E	E
	5.0	S	E	5.0	E	E	E	E	E	E	E	E	E
	7.5	E	E	7.5	E	E	E	E	E	E	E	E	E
	10.0	E	F	10.0	E	E	E	E	E	E	E	E	E
MN 1	2.5	B	B	2.5	A	B	B	B	B	B	B	B	B
	5.0	C	C	5.0	A	B	B	B	B	B	B	B	B
	7.5	C	D	7.5	A	B	C	C	C	B	B	B	B
	10.0	D	C	10.0	A	B	C	C	C	B	B	B	B
MN 4	2.5	C	D	2.5	A	B	B	B	B	C	C	C	C
	5.0	C	D	5.0	A	B	B	B	B	C	C	C	C
	7.5	C	F	7.5	A	B	B	B	B	C	C	C	C
	10.0	C	F	10.0	A	B	B	B	B	C	C	C	C

## REMARKS

- 1 12 cc of binder 'S' having a specific gravity of 1.3 gm/cc was mixed with 20 gm of coating powder.
- 2 Coated specimens were air dried for 6 hours at room temperature before exposure at high temperature.
- 3 In all cases the coating surface after 6 hours of air drying was even and showed no cracks, indicating good paintability and adequate adherence to the base.
- 4 In all cases the coating came off from the specimen surface by its self on cooling after firing at high temperature.
- 5 Surface appearance code :
  - (A) Practically no oxidation
  - (B) Slight oxidation without pitting
  - (C) Temper colours appeared on the surface without pit formation
  - (D) Light gray colour with slight pits formation
  - (E) Dark black coloured oxidation layer which came off as flakes
  - (F) Dark grey colour with heavy pitting.

reacting with the specimen surface. The coatings come off by itself because the coefficient of thermal contraction of the base metal and the coating was different. The metal contracts more rapidly than coating. So stresses develope in the coating and the bond between the specimen surface and coating breaks. Because of this the coating comes off by itself.

## CHAPTER IV

### CONCLUSIONS

The following conclusions can be drawn from the results which were shown in Chapter III:

1. Both coatings MN1 and MN4 are very effective in reducing the oxidation loss of mild steel and stainless steel. The percentage reduction in oxidation, in comparison to uncoated specimens, of mild steel and stainless steel is above 69% and 89% respectively at all temperatures and times. In case of tool steel coating MN4 is more effective than coating MN1 at all temperatures and times.
2. In case of mild steel and stainless steel the percentage reduction in oxidation with coatings MN1 and MN4 increased with time at all temperatures. But in case of tool steel at  $1100^{\circ}\text{C}$  the effectiveness of both the coatings MN1 and MN4 decreased with time.
3. For longer exposure the coating MN4 showed better protection to oxidation than coating MN1 in all steels.
4. The application of both the coatings is easy by brush painting. It would be possible to adjust the fluidity of the coating so that it could be applied by dip or spray coating methods also.

5. During cooling, after exposure at high temperature, both the coatings came off the specimen surface by itself.
6. Both the coatings showed good thermal conductivity.
7. Coating MN1 reduced the amount of decarburization and does not hinder the normal hardening treatment of steel. It is expected that coating MN4 will behave in a similar manner.
8. The surface covering power of the coatings is good. 20 gms of the coating powder mixed with 12 cc of the binder would cover about  $450 \text{ cm}^2$  surface area.
9. Coating MN1 and MN4 are cheap. Based on raw material costs only it would cost about Rs. 0.82 for coating MN1 and Rs. 0.74 for coating MN4 to cover about 1 Sq. Meter surface area.

REFERENCES

1. Metals Reference Book 5th Edition, Butterworths, London, 1976, pp. 1438.  
L
2. Metals Hand Book, Volume 2, 8th Edition A.S.M., Metals Park Ohio 1964.
3. A.G. Holchkiss and H.M. Webber 'Protective atmospheres' John Wiley and Sons, New York 2nd print 1963.
4. D.S. Clark and W.R. Varney 'Physical Metallurgy for Engineers' East-West Press Pvt. Ltd., 1968.
5. Y. Lakhtin, 'Engineering Physical Metallurgy' Mir Publications
6. Carl A. Zapfer, 'Sources of Hydrogen in Steel and Means for Its Elimination' Metal Progress, Vol. 43, No.3, March 1943, pp 397-401.
7. J.B. Seabrook, N.J. Grant and Dennis Carney 'Hydrogen Embrittlement of SAE 1020 Steel', Journal of Metals Vol. 188, No.11, Nov. 1950 pp. 1317-1321.
8. C. Burton Clark and C.L. Thomson 'Iron oxide conversion a Previously Unrecognized Course of Refractory Destruction' Industrial Heating Vol. 14, No.12, Dec. 1947, pp. 2056-58.
9. C.A. Leidholm 'High-Hydrogen Atmosphere Intensifies Quench Cracking Tendency' Metal Progress, March 1947, Vol.51, No.3, pp. 414-419.
10. Raymond. E. Birch 'Modern Blast Furnace Refractories' Blast Furnace and Steel Plant, Vol. 30, No.3, March 1942, pp. 345-350.
11. Foseco Trading A.G. Fr. 1, 512, 690 (Cl-C09d), 9 Feb. 1968, Chem. Abst. No. 99201W, Vol. , 1972.
12. Higuchi, Hisashi, Ghsuzu, Hirotoda, Uchino, Kaoru, Makijima, Hiroshi, Toyota, and Tsunehiko, Chem. Abst. No. 30035W, Vol. 83(4) 1975.

13. Popov, N.N., Chem. Abst. No. 34340U., Vol. 82(6), 1975.
14. Popov, N.N., Chem. Abst. No. 95393q, Vol. 81(16), 1974.
15. Popov, N.N., Chem. Abst. No. 125722c, Vol. 84(18), 1976.
16. Maragin, Yu. V., Arisemova I.V., Popov, N.N., Chem. Abst. No. 165302t, Vol. 85(22), 1976.
17. Popov N.N. Appen A.A., Antanova E.A., Chem. Abst. No. 24399d, Vol. 71(6), 1969.
18. Boggs, William E. Linderman, William A., Snow, Roland B. Chem. Abst. No. 174230n, Vol. 81(26), 1974.
19. Murav'ev, V.I., Chem. Abst. No. 49660z, Vol. 70(12), 1969.
20. Medvedev, G.A., Sokolova, N.I., Tsapalova, N.K., Chem. Abst. No. 128173s, Vol. 71(26), 1969.
21. Fujita Seichi, Chem. Abst. No. 94969v, Vol. 81(16), 1974.
22. Yu. V. Grdina, D.I., Tarasko and L.F. Shimova, Chem. Abst. No. 110845v, Vol. 67(24), 1967.
23. Colantuono William, R., Chem. Abst. No. 146581d, Vol. 78(8), 1973.
24. Masudo, Soichiro, Hasegawar, Kkirai, Fujita, Teruo, Shigashagida, Kazunaga, Chem. Abst. No. Vol. 85(26), 1976.
25. Matsuno, Fumio, Chem. Abst. No. 164784q, Vol. 85(22) 1976.